Gyrotron Processing of Materials

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Introduction

Microwave processing technology has taken on increasing significance in manufacturing. Numerous equipment configurations that take advantage of heating by coupling microwave radiation to materials have been developed for curing, bonding, and sintering materials. Such industrial microwave systems generally operate at centimeter wavelength frequencies of 0.915 or 2.45 GHz. Although they offer new methods of industrial processing, their application is often limited by the degree of coupling of the microwave energy to the materials to be processed.

Recently, millimeter wavelength generators have become available. They have enabled scientists and engineers to devise new methods for processing materials. Radiation in these superhigh frequency (SHF) domains interacts differently with materials than that at centimeter wavelengths. For many materials, the absorption of microwave energy at millimeter wavelengths is significantly greater than at 0.915 or 2.45 GHz. This in turn offers new opportunities for thermally processing materials in significantly shorter processing times, or for modifying their properties.

The recent collapse of the Soviet Union has opened previously closed laboratories and industrial facilities. Discoveries and equipment that were once closely guarded secrets are now being shown and shared. Institutes, which only a few years ago would have been suspicious of Western contacts now welcome potential investors, licensers, or co-workers. Some of these institutes have made significant advances in microwave instrumentation and its application to industrial processing.

The Institute of Applied Physics in Nizhny Novhorod (Gorky), Russia, has developed high-power gyrotrons that operate at millimeter wavelengths. The Salut Institute, also located in Nizhny Novhorod has developed prototypes of relatively compact and affordable gyrotrons that can operate at various millimeter wavelengths, in a continuous mode, and with power outputs up to tens of kilowatts. Energy conversion into microwaves is relatively efficient, up to 40%. These gyrotrons generate microwave energy in the form of a beam that can be focused, spread, rastered, or directed with metal mirror optics onto the material or part of the material to be processed.

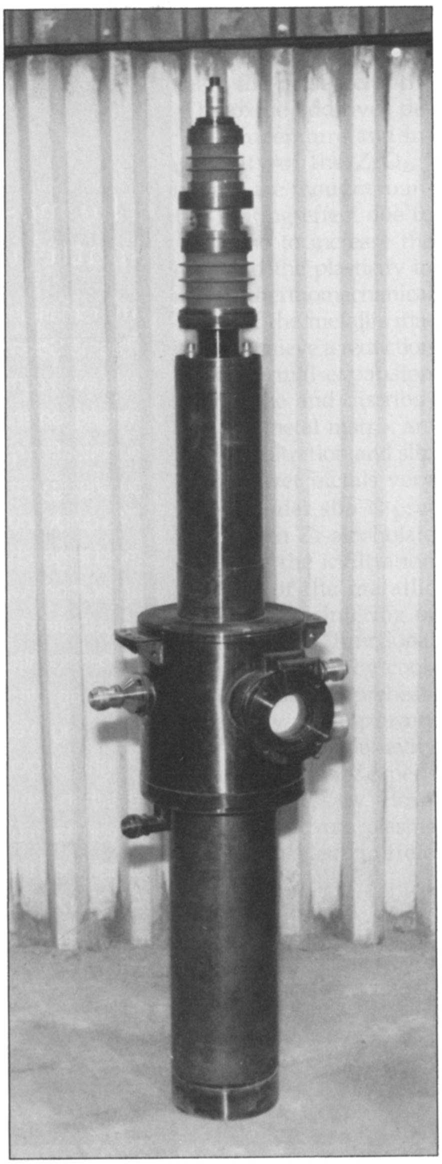

Figure 1. Gyrotron tube.

A photograph of a gyrotron tube is shown in Figure 1. The tube shell is constructed of stainless steel. High voltage is applied to the cathode, which is located at the top of the tube as shown (Figure 1); the window, through which the microwave beam exits the unit, is within the flange, facing the viewer, and is located just below the midpoint. The tube is approximately 165 cm long and weighs about 60 kg. During operation, the tube is mounted inside a shielding chamber and fits inside a superconducting magnet.

Scientists at the E.O. Paton Electric Welding Institute (PWI) in Kyyiv (Kiev), Ukraine, have developed numerous applications for this microwave tool. Some of these applications have been implemented in industrial production settings. Figure 2 shows a research gyrotron installation. Figure 3 shows the work chamber. The microwave beam enters the work chamber from the right and is deflected down onto the workpiece by the metal mirror mounted within the chamber. Shielding (removed for the photograph) and safety interlocks protect the operator. This article focuses on some of these new developments and opportunities in gyrotron microwave processing.

Properties of Gyrotron Microwave Radiation

The intense microwave beam generated by the gyrotron has unique properties. First, it is an efficient source for heating nonmetallic materials such as ceramics, glasses, polymers, and other organic substances. When such materials are exposed to gyrotron radiation, they can be heated to their melting points in seconds or even fractions of a second.

Second, in addition to heating materials very rapidly, gyrotron radiation heats the bulk of the material at a penetration depth of several millimeters to tens of millimeters. These penetration depths correspond to the dimensions commonly encountered during industrial processing. No other known heat source provides such heating. Lasers, plasmas, and infrared radiation penetrate only to the depth of a tenth or a hundredth of a millimeter, and all the material is heated from the surface. Centimeter microwave radiation

Figure 2. Research gyrotron installation.

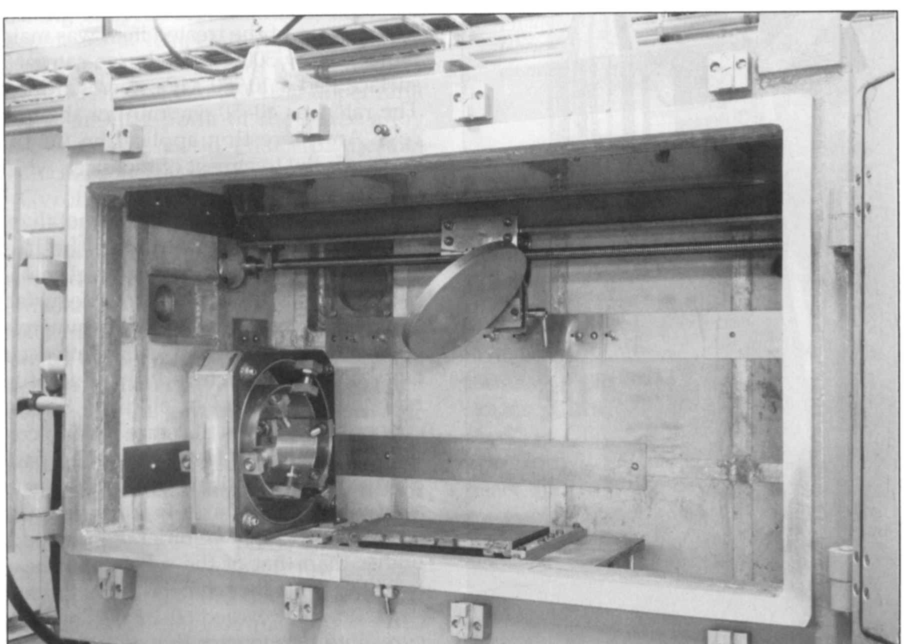

Figure 3. Gyrotron work chamber.

sources do not generate microwave energy as a beam. The material processed is "bathed" by the radiation, which penetrates deeply into the material with comparatively low interaction efficiency. Thus, gyrotron radiation can heat a cubic centimeter of aluminum oxide to its melting point in a fraction of a second; centimeter wavelength radiation generated by a magnetron or klystron will take tens of minutes to melt the same amount of material.

Third, gyrotron heating is selective. If two materials such as silicon carbide and quartz are subjected to gyrotron radiation, one will remain cold while the other will be heated to a high temperature. Since this heating is extremely fast, thermal conduction will not significantly raise the temperature of the nonabsorbing material, even if the two objects are in intimate contact.

These properties of gyrotron radiation, combined with the relative simplicity and reliability of the gyrotron equipment, make the gyrotron a promising tool for solving technical problems that were previously considered impossible to solve. Gyrotron applications can be broadly classified into three important areas: coating of materials, soldering and brazing, and treatment of polymers. Other significant applications that do not neatly fall within these broad classifications include the treatment of semiconductors and the synthesis of materials.

Coating of Materials

The corrosion resistance of pipes and other metal objects can be significantly improved by coating them with a polymer. A widely used method for applying such coatings is as follows. First, the metal surface is heated to 140–160°C, and then a two-layer film consisting of polyethylene and a primer layer is pressed onto the surface. The hot metal heats the polymer/primer film, bonding it to the metal. The degree of adhesion is proportional to the temperature—the higher the temperature during bonding, the better the adhesion. It is undesirable, however, to increase the bonding temperature beyond 160°C because the polymer itself will be damaged.

Gyrotron microwave radiation has been used to heat the interface between a metal and polyethylene to form a stable, strong bond between the polyethylene and the metal surface. This coating technique relies on three components, each of which interacts differently with the radiation. The first component, the metal, is the microwave reflecting surface. The second component, the primer, is designed so that it absorbs microwave radiation well and adheres to both the metal and the polyethylene. The third component is the polyethylene, which has low microwave radiation absorption. When this system is subjected to gyrotron radiation, the primer layer heats very rapidly. Since the polymer is relatively transparent and the metal is reflective, only the primer layer is heated. In actual applications, the temperature of the primer layer was raised to more than 500°C while the temperature of the polyethylene remained below 100°C. Thus, heat can be selectively applied only to the middle layer of a three-layer structure. Protective coatings applied this way have an adhesion that is several times stronger than that of conventionally applied coatings. Furthermore, no other existing technology has produced coatings that match the excellent water resistance of coatings applied using gyrotron heating. This process has been used commercially to protect large-diameter oil pipes from corrosion. The protective layer was applied at more than 100 m^2/h at the installation shown in Figure 4.

This coating technique is not limited to applying polyethylene. It can be used to apply materials such as Teflon, polypropylene, and other nonpolar polymers that are relatively transparent to gyrotron radiation. Its applications include protective coatings on metals that must function in corrosive environments, for example, in chemical handling equipment and structures exposed to salt water.

This ability of gyrotron radiation to selectively heat materials can be used to form a ceramic glaze with a wide transition zone between it and a substrate such as firebrick. A ceramic glaze powder is placed on the firebrick surface and exposed to a gyrotron microwave beam. Initially, the gyrotron radiation heats all the ceramic material (powder and firebrick), creating a small thermal gradient in the system. As the exposure continues, the thermal gradient increases until the surface of the firebrick melts. At this point, the molten surface becomes reflective and redirects the gyrotron beam back through the ceramic powder above it. The beam passes through the ceramic powder twice, causing it to melt. The resultant coating layer has a wide transition zone, strong adhesion, and high resistance to thermal shock. Such coatings can be 10 mm or more thick with a transition zone several millimeters thick.

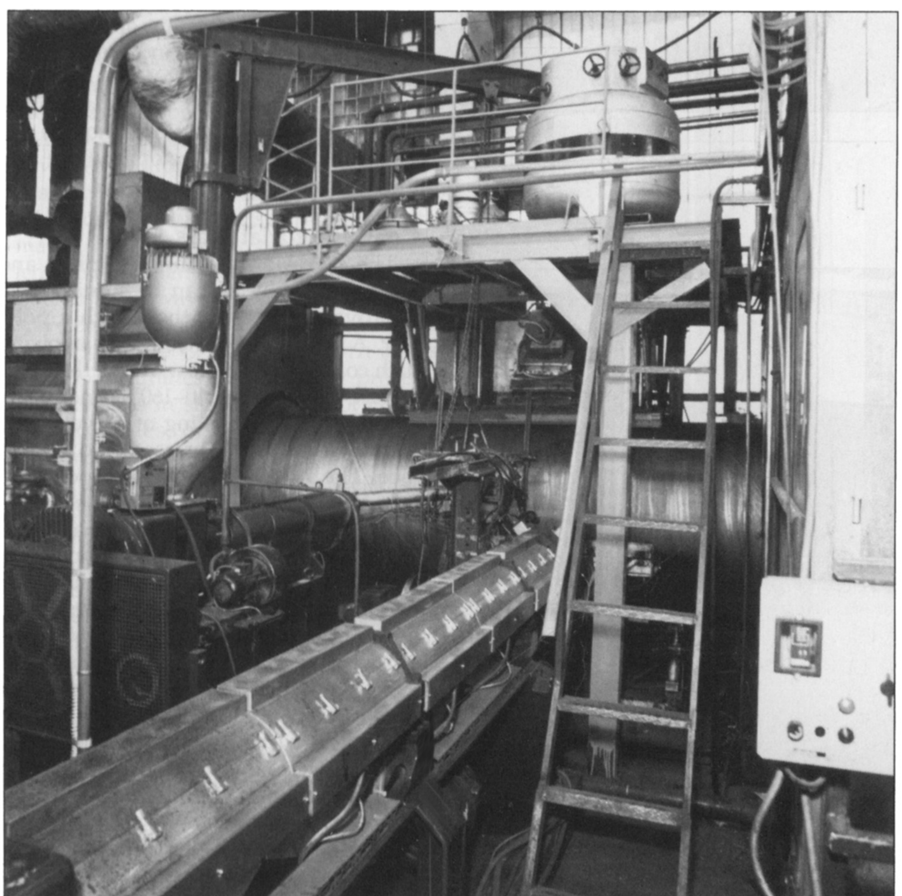

Figure 4. Commercial large-diameter pipe coating installation that uses a gyrotron.

These characteristics yield excellent performance conditions under cyclical mechanical and thermal loads. This type of coating has been used for steel casting ladle linings, gates, and pipes for continuous steel casting. The service lifetimes of gates coated using gyrotron microwave processing techniques are several times better than those of conventionally prepared gates. In another application, refractory crucibles were coated with a 5–6 mm layer of yttrium oxide and used to 2400°C to melt highly reactive metals.

As noted previously, metals reflect gyrotron radiation and do not interact with it. It is possible, however, to use gyrotron radiation to treat metals. One such application is metal surface hardening. For example, a tool to be hardened is first coated with a thin layer of a powder or paste containing the hardening components. Gyrotron radiation then rapidly heats only this covering layer to a very high temperature, causing diffusion from the paste into the metal. This process takes advantage of the high diffusion rates possible only at very high temperatures, but it accomplishes this without heating the entire tool. In one case, steel was diffusion hardened with boron using a paste containing B_4C. Gyrotron radiation heated the paste to 2450°C, while the average temperature of the treated item was maintained below 500°C. The boron-saturated surface had a 10,980 MPa microhardness. The rate was 40–50 cm^2/min for this process. An interesting application for this process is the treatment of tool steels which cannot be heated above 600°C.

Dielectric materials can be metalized using gyrotron radiation as the heat source. In this process, a special metal-based paste is fused or cured on the surface of the dielectric substrate. A commercial application of this process is printed circuit board manufacture using thick-film technology instead of etching. Special copper-based pastes that cure to form conductors have been formulated. The paste is screened onto a fiberglass laminate substrate. Since the paste is formulated to have an absorption an order of magnitude higher than that of the substrate, it heats to 240°C while the temperature of the substrate does not exceed 60–80°C. The paste cures into a conductor that adheres to the substrate. The solderability, adhesion, conductivity, and other parameters of the cured conducting lines meet thick-film technology requirements. Circuit board conducting lines fabricated by using this process are shown in Figure 5. Processing equipment has been designed for continuous mode operation in an industrial setting, with 10 m^2/h throughput. With this process, circuit boards can be fabricated without using etchants or other environmentally hazardous materials.

On ceramic substrates, the copper paste can be fused so quickly with gyrotron radiation that the copper does not oxidize. As a result, no shielding gases are required. The adhesion of the copper to the ceramic substrate is 150 kg/cm^2, which is two to three times that achieved with the conventional burn-in methods. The copper layers are highly resistant to water. A microcircuit board fabricated this way is shown in Figure 6.

The examples just discussed do not cover all the possible ways that this new tool can be used to apply coatings to materials. Other possible applications include the deposition of superconducting films, vacuum deposition of superpure and refractory coatings, application or repair of enamel coatings on metals, and others.

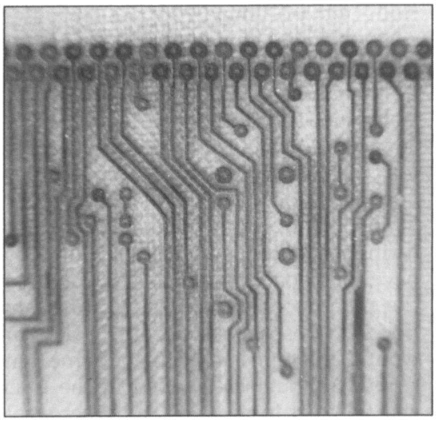

Figure 5. Fiberglass laminate circuit board fabricated using a gyrotron radiation absorbing metal-based paste.

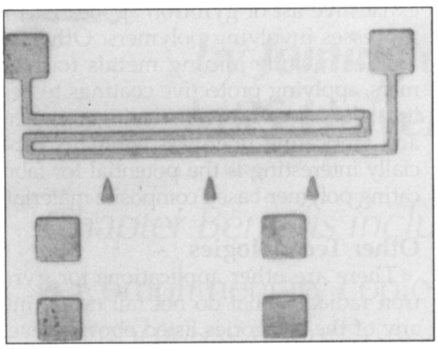

Figure 6. Ceramic microcircuit board fabricated using a gyrotron radiation absorbing metal-based paste.

Soldering and Brazing

Industrial tool applications often require affixing synthetic superhard materials such as diamond or cubic boron nitride (CBN) to a metal tool. Numerous alloys have been developed for this task. The mechanical bonding properties of brazing alloys tend to improve with their melting temperature. Diamond, however, graphitizes at temperatures above 800°C. Similarly, CBN transforms to the hexagonal phase. This sets a practical upper limit to the melting point of the braze alloy and, therefore, to the choice of the mechanical properties of the bond.

Gyrotron processing offers a unique way to get around this limitation. Diamond and CBN are transparent to gyrotron radiation. The brazing alloy can be formulated into a paste that absorbs millimeter microwave radiation. A layer of the brazing alloy paste is placed between the superhard material and the tool. When subjected to the gyrotron beam, the brazing paste absorbs most of the energy and melts in just a few seconds. The gyrotron heating takes place so quickly that there is insufficient thermal transfer to the diamond or CBN for the phase transformation to take place. In just a few seconds the synthetic superhard material is brazed onto the tool. Diamonds and CBN crystals have been brazed to tools with pure copper, and cobalt- or nickel-based alloys that have melting points higher than 1000°C without damage to the diamond or CBN. This technology allows the selection of a brazing alloy that optimizes the desired physical and mechanical characteristics of the tool.

An analogous process can be used to achieve low-pressure compaction of diamonds or CBN using high-temperature brazes. In this case, small crystals of diamond or CBN are mixed with a brazing alloy powder. The gyrotron radiation heats the brazing alloy extremely rapidly to a temperature above the diamond or CBN phase transition temperature while low pressure is applied. Again, since the heating is very fast and the exposure time very short, the diamond or CBN does not undergo a phase transformation. Since the compaction can take place at low pressure, large pieces can be easily and inexpensively fabricated using high-temperature binders.

Likewise, ceramic articles can be joined using high-temperature brazes. The items to be joined are assembled with the brazing materials between them. Under gyrotron radiation, the highly absorbent braze creates a locally heated region at the joint. Because of this unique heating method, the melting point of the braze can actually be higher than the permissible temperature of the joined ceramic material. In tests, the temperature at the joint exceeded 1600°C, but the surrounding ceramic material was not heated significantly. Since heating takes place rapidly, and the heating time is short, there is insufficient heating by thermal conduction to overheat the surrounding material. This joining process can be used to fabricate complex shapes that must operate at high temperatures, such as turbine blades or engine components.

Gyrotron radiation can be used to mass solder electronic components onto ceramic or fiberglass printed circuit boards. In this process, the solder paste is screened onto the regions to be soldered, and the board is exposed to gyrotron radiation. The radiation selectively heats the paste without significantly heating the board or components. This process can be used for surface-mounted or lead-type components.

Undoubtedly, there are many other material/braze and material/solder systems that can take advantage of the unique heating capabilities of gyrotron radiation.

Treatment of Polymers

Polymers can be selectively and rapidly heated by gyrotron radiation. This effect can be used to hermetically seal electronic components. Often a polymer forms the hermetic seal between the wire lead and component body. The sealing time is set by the cure time of the polymer, which in turn depends on temperature. A high-temperature cure causes the air trapped inside the heated component to expand and form pores in the seal. A low-temperature cure process, on the other hand, takes a long time. With gyrotron radiation, heat can be applied directly and exclusively to the sealing polymer. The component remains cool, pores are not formed in the seal, and the total cure time is reduced to only a few seconds.

Gyrotron radiation can also be used to manufacture fiberglass reinforced polymer laminate sheets. In conventional manufacturing processes, a fiberglass mesh is passed through a tank of polymer dissolved in a solvent. The polymer solution impregnates the mesh, which is then dried in large ovens. The fiberglass/poly-

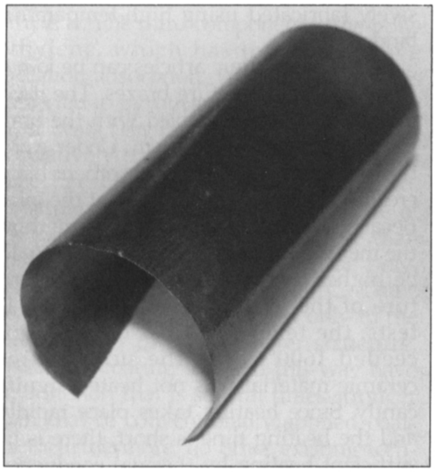

Figure 7. Polymer-impregnated fiberglass sheet fabricated using gyrotron radiation heating.

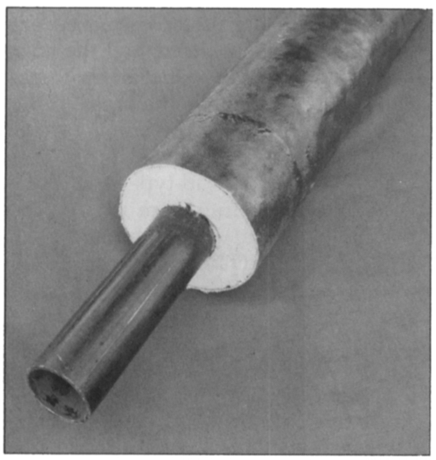

Figure 8. Pipe with insulation protected by a polymer layer cured with gyrotron radiation.

mer composite is then cut into sheets, stacked, and fused into multilayered sheets. A new process, based on gyrotron heating, eliminates the solvents and large drying furnaces. A fiberglass mesh is covered with a thin layer of polymer powder. Gyrotron radiation selectively heats and melts only the polymer just as it passes between a pair of opposing metal rollers. Since the rollers are metal, they reflect the microwave radiation and the heat is concentrated in the area between the rollers. As the mesh with the molten polymer passes through the rollers, they force the liquefied polymer into the fiberglass mesh. Since the metal rollers are not heated by the gyrotron radiation, they can be kept cool. This prevents the molten polymer from adhering to them. The cured sheet exits the rollers and is ready for the next manufacturing step. A sheet fabricated this way is shown in Figure 7.

The gyrotron can be used in manufacturing reinforced polymer structures that have rotational symmetry. For example, in the manufacture of fiber-reinforced cylindrical vessels, reinforcing fibers are wound, impregnated with a polymer, and cured. Air bubbles trapped in the fibers and polymer form pores and other weakening defects during the polymerization process. Gyrotron radiation can rapidly heat and cure successive layers as they are wound, preventing air from getting trapped. Since gyrotron heating is localized, selective, and very fast, curing can occur as the part is manufactured.

Again, the examples just cited are not an exhaustive list of gyrotron applications in processes involving polymers. Other examples include joining metals to polymers, applying protective coatings to heat insulators (see Figure 8), drying polymers, and fabricating fiberglass laminate. Especially interesting is the potential for fabricating polymer-based composite materials.

Other Technologies

There are other applications for gyrotron radiation that do not fall neatly into any of the categories listed above. Nevertheless, they are very interesting and we will describe some of them.

The selective absorption of gyrotron radiation can be used to synthesize materials that are expensive to synthesize with alternative processes. For example, Si_3N_4 can be formed by heating elemental silicon in a nitrogen atmosphere in a plasma or other furnace. There is an alternative reaction for forming Si_3N_4:

$$SiC + O_2 + N_2 \rightarrow Si_3N_4 + CO_x \uparrow \text{ (1600°C)}$$

Although SiC is less expensive than elemental silicon, this reaction cannot be implemented in practice. This reaction takes place at 1600°C, but in the presence of oxygen the formed Si_3N_4 immediately oxidizes. Gyrotron radiation heating could be used to heat the SiC without oxidizing the Si_3N_4. SiC is a good absorber of microwave radiation, but Si_3N_4 has absorption two orders of magnitude lower. When the SiC is exposed to the gyrotron beam, it heats rapidly and converts to Si_3N_4. As soon as the SiC is converted to Si_3N_4, however, the microwave absorption and resultant heating automatically stops. The whole reaction takes place in a few seconds. X-ray diffraction analysis shows that two types of Si_3N_4 powders can be produced by this process. The first consists of pure Si_3N_4; the second consists of a core of SiC with a surrounding Si_3N_4 layer.

A totally new application for gyrotron microwave radiation lies in semiconductor processing. Abnormally fast diffusion of different species is observed when a semiconductor material is exposed to gyrotron radiation. Diffusion processes take place rapidly, in only tens of seconds and sometimes even in fractions of seconds. Particularly noteworthy is that the semiconductor itself remains at a relatively low temperature. Comparable diffusion by thermal processes takes many hours and requires very high temperatures, which can generate thermal defects in the semiconductor material. Selective fast diffusion by gyrotron radiation can be used to homogenize semiconductors, activate dopants, form ohmic contacts, and more. Some preliminary results indicate that subjecting a finished semiconductor component to gyrotron radiation improves the operating characteristics.

Gyrotron radiation allows the high-temperature ($T > 2000$°C) treatment of ceramic materials. Heating can be accomplished by directly exposing the ceramic material to gyrotron radiation, or it can take place in ovens that are specially constructed to accommodate a gyrotron. It has been observed that many ceramic refractory powders change their properties if subjected to high-temperature anneals. Components made of these refractory ceramic powders have improved operating characteristics.

Conclusions

Our examples present a partial overview of materials processing applications that have been devised for the gyrotron. This new manufacturing tool offers new ways of controllably and rapidly delivering heat selectively to the desired part of a multicomponent system. The success of any application, however, depends on the proper choice of materials, or combinations of materials, based on their SHF microwave characteristics. These characteristics must be considered in conjunction with other design parameters such as mechanical and functional ones and, most importantly, the relative cost of alternative technologies. Gyrotron microwave processing should not be considered simply as a faster version of lower frequency microwave processing or other heating techniques. Its true value as an industrial tool is based on an ability to devise new engineering solutions previously considered impossible or impractical. Gyrotron processing provides a new level of control to the process designer.

Acknowledgments

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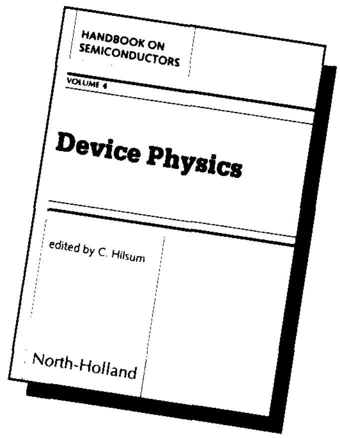

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