# MECHANICAL PROPERTIES OF COMPOSITE BASED THERMAL BARRIER COATING FOR AEROSPACE APPLICATION, SINGLE AND DOUBLE CERAMIC LAYER COATINGS

**PROJECT III REPORT**
**(18MPR804)**

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***In partial fulfillment for the award of the degree***

***of***

**BACHELOR OF ENGINEERING**

***in***

**MECHANICAL ENGINEERIG**


**GOVERNMENT COLLEGE OF ENGINEERING, BARGUR.**

**(An Autonomous Institution Affiliated to Anna University-Chennai)**


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# GOVERNMENT COLLEGE OF ENGINEERING,BARGUR.

**(An Autonomous Institution Affiliated to Anna University-Chennai)**

## BONAFIDE CERFICATE

Certified that this project report **"MECHANICAL PROPERTIES OF COMPOSITE BASED THERMAL BARRIER COATING FOR AEROSPACE APPLICATION, SINGLE AND DOUBLE CERAMIC LAYER COATINGS"** is the Bonafide work of

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**INTERNAL EXAMINER** **EXTERNAL EXAMINER**

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# ABSTRACT

Lanthanum zirconate is a promising candidate material for thermal barrier coating (TBC) applications due to its low thermal conductivity and high temperature phase stability. However, its application is limited by thermal durability caused by low fracture toughness and low coefficient of thermal expansion And Minimum porosity level And maximum hardness . We recently developed LZ/YSZ composite TBC systems using blended LZ and YSZ powders, which have demonstrated excellent thermal cycling performance. In this study, the mechanical properties of the composite TBCs were characterised using both indentation and Vicker's hardness tests. The indentation results show that both Young's modulus and hardness increase with increasing YSZ content, suggesting the mechanical properties can be tailored by changing the volume ratio of YSZ. The hardness results show the same dependence with YSZ content, which is confirmed by the analytic models based on composite theory.

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# CHAPTER 1

# 1 INTRODUCTION

## 1.1 Thermal barrier coatings

Thermal barrier coatings (TBCs) have been extensively used in gas turbines, aircraft and marine propulsion systems to provide thermal insulation to metallic components from hot and corrosive gases [1,2]. A typical TBC is composed of four layers of materials, including (1) a substrate of nickel- or cobalt-based super alloy, (2) an oxidation-resistant metallic bond coat of NICrAlY, (3) a thermally grown oxide (TGO) layer and (4) a ceramic top coat. Eight wt-% yttria-doped stabilised zirconia (8YSZ) is the currently used standard TBC material due to its relatively low thermal conductivity, high coefficient of thermal expansions , good mechanical properties and chemical inertness in gas turbine operating conditions [3–5]. TBCs are coated using either air plasma spray (APS) or the electron-beam physical vapour deposition process, in which coating materials are additively deposited on the component surfaces. Development of advanced TBC materials for advanced gas turbines operated at high temperatures has been an active research area. In general, there are two ways to enhance TBC's performance via reducing the thermal conductivity:

(1) feedstock powders with intrinsic low thermal conductivity,

(2) control of top coat's porosity.

Recently, a new promising TBC material, lanthanum zirconate (La2Zr2O7, LZ) was proposed, which has a pyrochlore structure. Compared with YSZ, LZ has a lower intrinsic thermal conductivity, better high temperature phase stability (up to testing temperature 1400°C) and higher sintering resistance [6–8]. However, due to its low coefficient of thermal expansion, high thermal stresses occur near the interface between the top and bond coats, causing delamination and low durability in cyclic thermal environments. Double-ceramic-layer (DCL) TBCs have been proposed, which include a top ceramic layer and a thin ceramic buffer layer. In the DCL TBCs, the top layer is made by low thermal conductivity LZ material, which offers good heat insulation. The buffer layer is usually made by the traditional TBC materials, usually YSZ, which provides CTE mismatch needs with the bond coat. The buffer layer also can be designed with the DCL TBC as a stress buffer layer, to reduce the stress level and ensure thermal durability [12–15]. Through the design of experiment approach, we have identified DCL TBC composed of LZ top layer and porous YSZ buffer layer has low thermal conductivity and good thermal cycling durability, compare able to pure YSZ coating [12].

- Resistance to wear, abrasion and erosion.
- Thermal barrier coating to protect structures and materials.
- Corrosion resistance in air and marine environments.
- Protection against high temperature oxidation, erosion and corrosion.
- Electrical resistance, electrical conductivity, or electro-magnetic shielding.
- Layer-by-layer manufacturing of shaped component.
- Dimension restoration for worn surfaces.
- Building composite structures of metals and ceramics.
- Adhesive base for bone in growth in medical implant

Although the initial success, there is a strong motivation to further increase the durability of the TBCs in thermal cycling conditions, in order to meet the requirements of advanced gas turbines. To this end, we recently proposed a new coating architecture with a composite microstructure. The new composite microstructural design employs the blended powders of LZ and YSZ with varying volume ratios. The composite approach is fundamentally different from previous layered microstructures which used single material or same material with varying densities in each layer. Using the new microstructural design, we have demonstrated that the LZ/YSZ TBC systems have a much better thermal cycling performance than the layered coatings, using a variety of thermal cycling tests, including furnace cyclic thermal fatigue, thermal shock and jet engine thermal shock [14]. In this study, we focus on the mechanical properties of the new composite LZ/YSZ TBC systems, to complement our previous thermal cycling data. The mechanical properties of the as-sprayed TBC specimens were measured using both indentation and hardness tests. Analytic models of Young's modulus at different LZ/YSZ ratios were employed based on composite theory. The predicted Young's modulus values were compared with the experimental measurements.

Because protective coatings are becoming a widespread part of metal working,thermal spray is a rapidly growing market.

The methods classified under thermal spraying include flame spraying, plasma spraying, High-Velocity-Oxyfuel (HVOF), and twin-wire-arc spraying, all of which share the following common features:

1. Production of molten particles: molten particles are formed in the spraying gun region.
2. Transport of molten particles: molten particles are propelled towards the substrate top coated.
3. Particle deposition: molten particles are deposited and flattened on the substrate,forming solidified or sintered splats that form the coating layer.

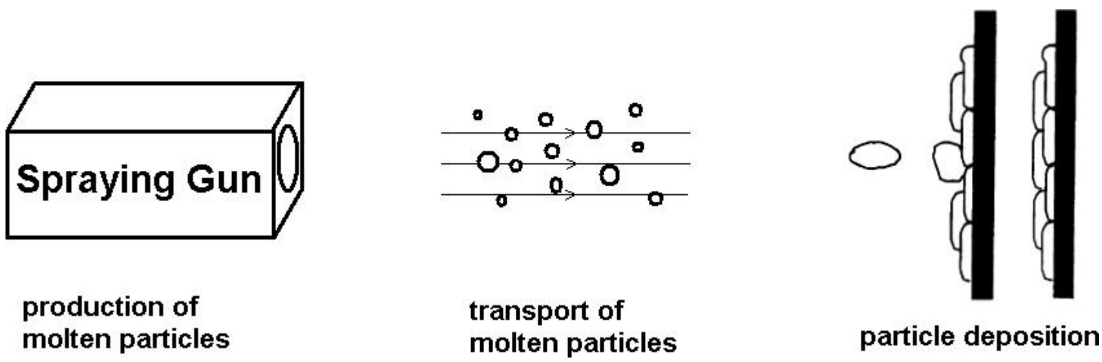


**Fig 1.1 Basic principles underlying the thermal spray processes: Production, Transport, and Deposition of molten particles.**

## 1.2 Development of LZ powder and its characterization

LZ powder is a type of energetic material that has been developed for various applications, including military and civilian use. The development of LZ powder involves a series of processes, including synthesis, purification, and characterization.

Synthesis: LZ powder is typically synthesized using a two-step process. The first step involves the preparation of the precursor materials, which are then subjected to a thermal treatment to yield the final product. The precursor materials are usually a mixture of metal powders and fuel, which are reacted together in a controlled environment to form the desired product.

Purification: The synthesized LZ powder is then purified to remove any impurities or unwanted compounds that may have been formed during the synthesis process. This is typically achieved through a series of chemical treatments or physical separations, such as filtration or centrifugation.

Characterization: After purification, the LZ powder is characterized to ensure that it meets the desired specifications for its intended use. This involves a range of analytical techniques, such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermal analysis, which provide information about the physical and chemical properties of the powder.

The development of LZ powder continues to be an active area of research, with ongoing efforts to improve its properties and explore new applications. Some of the key areas of focus include enhancing its energy density, improving its stability and safety, and exploring novel synthesis methods.

## 1.3 Development of YSZ powder

YSZ powder, also known as yttria-stabilized zirconia powder, is a ceramic material that has gained significant attention due to its unique properties and various applications in a wide range of industries.

The development of YSZ powder can be traced back to the 1960s when researchers discovered that the addition of yttria to zirconia could improve its stability at high temperatures. Since then, the synthesis of YSZ powder has undergone several improvements to enhance its properties and increase its commercial viability.

One of the most commonly used methods for the production of YSZ powder is the solid-state reaction method. In this process, zirconia and yttria powders are mixed together and heated at high temperatures to form YSZ. The purity of the starting materials and the calcination conditions play a crucial role in determining the quality of the final product.

Another method for the synthesis of YSZ powder is the sol-gel method, which involves the formation of a colloidal suspension of metal oxide precursors that are then converted to YSZ through a series of thermal treatments. This method allows for the production of high-purity YSZ powder with excellent homogeneity and controlled particle size.

YSZ powder finds a wide range of applications, including as a material for solid oxide fuel cells, thermal barrier coatings, oxygen sensors, and dental implants. Its high ionic conductivity, thermal stability, and bio compatibility make it an attractive material for these applications.

## 1.4 Plasma spray coating

Plasma spray coating is a commonly used technique for applying thermal barrier coatings (TBCs) to metal components. It involves using a high-temperature plasma arc to melt and propel ceramic or metallic particles onto a surface, where they solidify to form a coating.

The process begins with the preparation of the metal substrate, which is typically cleaned and roughened to promote adhesion. The ceramic or metallic powder is then fed into a plasma spray gun, where it is heated to a high temperature by a plasma arc. The molten particles are propelled towards the substrate, where they impact and solidify, forming a dense and uniform coating.

The properties of the plasma spray coating can be tailored by adjusting the spray parameters, such as the particle size, velocity, and temperature. This allows for the creation of coatings with specific characteristics, such as porosity, thickness, and adhesion strength.

Plasma spray coating is widely used in a variety of industries, including aerospace, power generation, and automotive, due to its ability to produce high-quality, durable coatings that protect metal components from high temperatures and corrosive environments.

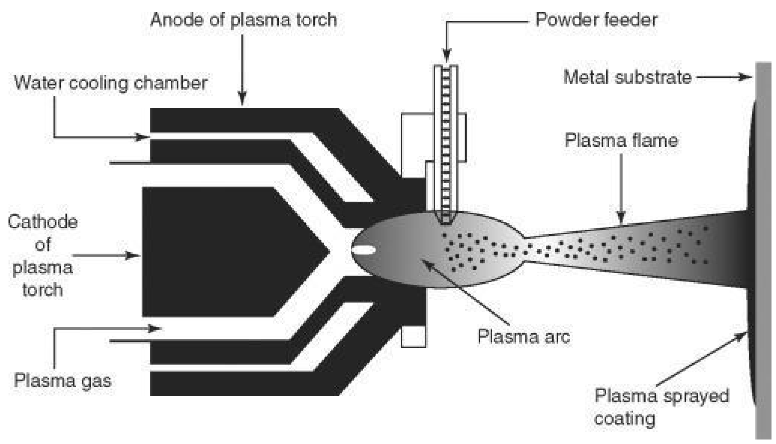


**Fig 1. 2 Schematics of Plasma spray coating and its major components**

## 1.5 Nickel Chromium Alloys

Nickel chromium alloys, also known as Ni chrome alloys, are a group of alloys that are used for their excellent high-temperature strength and resistance to oxidation and corrosion. These alloys consist of mainly nickel and chromium, with small amounts of other elements such as iron, silicon, and aluminum.

Chromium is added to nickel alloys to increase their resistance to oxidation and corrosion, as chromium forms a protective oxide layer on the surface of the material, which prevents further oxidation. The addition of other elements to nickel-chromium alloys can also improve their mechanical properties, such as strength, ductility, and toughness.

One type of nickel chromium alloy is the Ni chrome 80/20 alloy, which contains 80% nickel and 20% chromium. This alloy is commonly used in heating elements, resistance wires, and thermo couples due to its high-temperature strength and resistance to oxidation and corrosion.

Another type of nickel chromium alloy is the Kanthal A1 alloy, which contains 22% chromium, 5% aluminum, and the remainder iron. This alloy is used in heating elements, furnace components, and thermocouples due to its high-temperature strength and resistance to oxidation and corrosion.

Nickel chromium alloys are commonly used in high-temperature applications such as furnaces, heaters, and ovens, as well as in the aerospace, automotive, and chemical industries. They are also used in the production of resistance wires for electronic cigarettes and other electronic devices.

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## 1.6 Inconel 718

Inconel 718 is a high-strength, corrosion-resistant nickel-chromium alloy that is used in a wide range of high-temperature applications. It was first developed in the 1960s by Special Metals Corporation and has since become a widely used material in the aerospace, nuclear, and chemical industries.

The composition of Inconel 718 includes 50-55% nickel, 17-21% chromium, 4.75- 5.5% niobium, 3-5% molybdenum, and smaller amounts of other elements such as iron, titanium, aluminum, and copper. The addition of niobium and molybdenum to the alloy enhances its strength and makes it resistant to corrosion and oxidation at high temperatures.

Inconel 718 has excellent mechanical properties, including high tensile and yield strength, good fatigue resistance, and excellent toughness. It is also highly resistant to cracking and stress corrosion, making it suitable for use in harsh environments.

In the aerospace industry, Inconel 718 is used in the manufacture of aircraft engine components such as turbine blades, discs, and casings, as well as in rocket motors, and space vehicles. It is also used in the oil and gas industry for wellhead and downhole components that require high strength and corrosion resistance at high temperatures.

The production of Inconel 718 involves several processes, including melting, casting, forging, and heat treatment. The material is usually produced in the form of billets, bars, sheets, and plates, which are then machined into the desired shape.

# CHAPTER 2

## LITERATURE REVIEW

**Jing Zhang (2020)** Lanthanum zirconate ($La_2Zr_2O_7$) has been proposed as a promising thermal barrier coating (TBC) material due to its low thermal conductivity and high stability at high temperatures. In this work, both single and double-ceramic- layer (DCL) TBC systems of $La_2Zr_2O_7$ and 8 wt.% yttria-stabilized zirconia (8YSZ) were prepared using air plasma spray (APS) technique.

**S. Karthikeyan (2019)** Plasma-sprayed yttria-stabilized zirconia (YSZ) coating has been considered to be a good protective coating material for high-temperature applications on account of its superior properties and life cycle costs. However, thermal barrier coatings (TBCs) have engineering reliability problems in tailoring the microstructure and mechanical properties towards achieving both prime reliance and manufacturing reproducibility.

**Wagner M et al., (2021)** recommended for the use of the twin wire arc spraying process for fabricating high-density steel coatings at high deposition rates, highcoating density, uniform hardness across the coating, and good adhesion to the substrate.

**Palma De et al., (2022)** investigated changes in mechanical properties appear to correlate to microstructural changes that relate to the oxide content, porosity content, and mixing of the phases, though a larger sample size would be needed to confirm these relationships. Bend testing yielded an average in-plane moduli of the substrate modulus,depending on spray conditions, and average strength of the coating

**Liu H et al., (2010)** studied that, the increased hardness and corrosion resistance of the composites deposited by TWAS in a single step, have important consequences to revolutionize the cost-effective protective coatings suitable for onsite processing

**Chen Y et al., (2019)** investigated Fe-Al composite coating was manifested that metallurgical reaction is rarely generated between the anode and cathode for the hybrid twin-wire arc spraying. A mechanically mixed coating is therefore formed, in which the aluminium splats and carbon steel splats are inter-deposited.

**Yao et al., (2019)** studied the application of FeCrNbBSiC coating not only increases surface temperature of piston as improving power efficiency but also provides an additional protection without sacrificing the strength of aluminium alloy substrate. Significant temperature reduction by traversing this coating reveals its excellent thermalbarrier ability.

**Kim et al., (2015)** studied that TWAS process can be effectively utilized forengine applications in conjunction with optimum honing process for aluminium alloy cylinder liners. In this regard, conventional cast iron cylinder liners may be replacedwith lighter. TWAS coated aluminium alloy cylinder liners without compromising theirTribological performance and manufacturing cost.

**Horner et al, (2015)** studied a design of experiments approach to describe process parameter. Process relationships were investigated with the intent of maximizing or minimizing each coating property. It was determined that spray pattern area was most affected by primary gas pressure and secondary gas pressure. Pattern eccentricity was most affected by secondary gas pressure. The result revealed that the Coating deposition rate was most affected by arc current.

**Morquecho et al, (2014)** investigated results that indicates the twin wire arcspraying is a promising technique to prepare amorphous crystalline coatings. The use ofthe arc thermal spraying process enables use of a commercial substrate such as AISI 1018 steel for applications requiring high hardness and low wear.

**Bansala et al, (2020)** investigation shows that the rise in ZnO content in pure HA coatings, the surface properties as well as the corrosion resistance improved significantly. The contact angle of substrates under examination exhibits hydrophilic nature. Collectively, the findings of this study signify HA/ZnO reinforced coatings is a promising approach to improve surface properties and corrosion behaviour of Magnesium alloys for future bone implant applications.

**Nur Suhaili et al. (2022)** effect of stand-off distance on the microstructural, mechanical and physical properties of al-Zn pseudo-alloy coating prepared via wire arcspray process. The stand-off distance (SOD) parameter was varied at 100, 150, 200, 250, and 300 mm. The effect on the properties of developed coating layer on microstructure, thickness, hardness, roughness, and adhesion was investigated.

# CHAPTER 3

## 3.1 OBJECTIVE AND METHODOLOGY

- The objective of a coating process is to apply a thin layer of material, known as a coating, onto the surface of a substrate for a variety of reasons such as protection, decoration, or functionality.
- Studying the effects of plasma sprayed thermal barrier coating process parameters on porosity level and hardness.
- Analysing and discussing the experimental results.

## 3.2 Work Plan

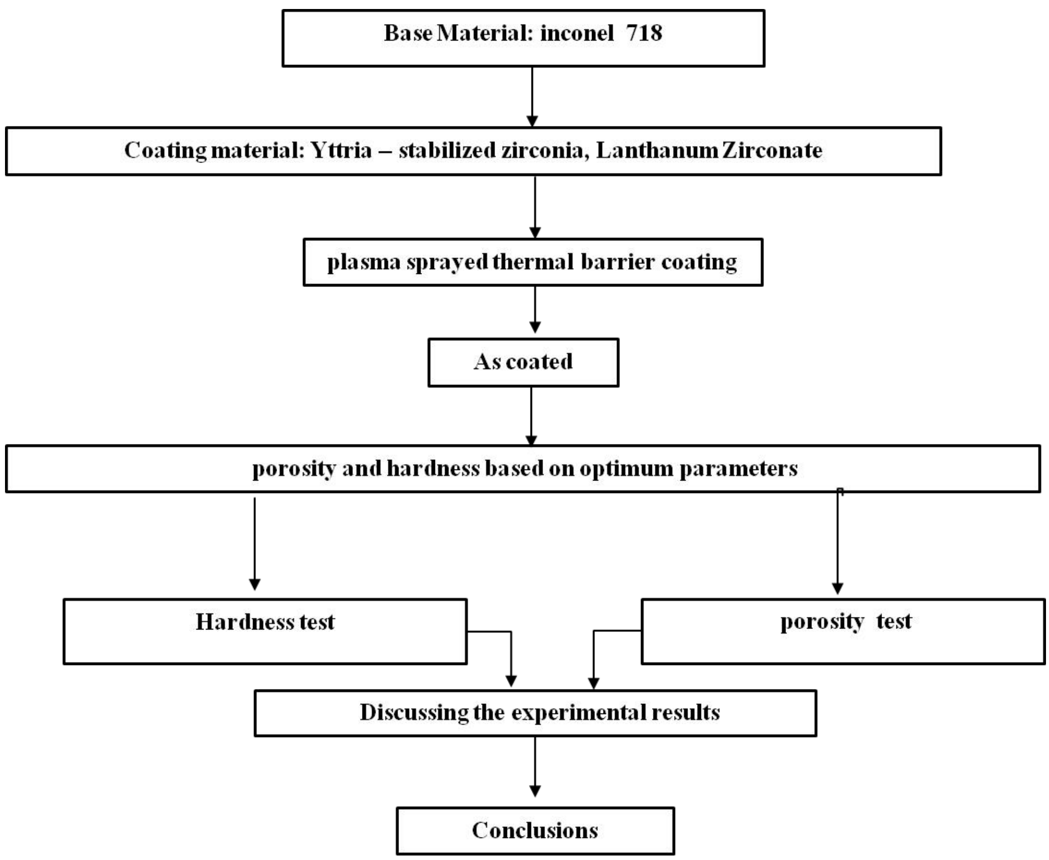


**Fig 3.1 work plan**

## 3.3 Chemical Composition Of Inconel 718

| Element | Content % |
|---|---|
| Nickel | 50.0 up to 55.0 |
| Cobalt | 1.00 |
| Chromium | 17.00 up to 21 |
| Molybdenum | 2.80 up to 3.30 |
| Iron | 17.0 |
| Silicon | 0.35 |
| Carbon | 0.08 |
| Aluminium | 0.60 |
| Titanium | 0.90 |
| Copper | 0.30 |

**Table 3.1 Chemical Composition Of Inconel 718**

# CHAPTER 4

## 4.1 Metallographic Preparation - Rough Grinding

Belt grinding is an abrasive machining process used on metals and other materials. It is typically used as a finishing process in industry. A belt, coated in abrasive material, is run over the surface to be processed in order to remove material or produce the desired finish.

**Figure 4.1 A sketch of the rough grinding machine, which is available in Government College of engineering, Bargur.**

## 4.2 Working Principle

The abrasive belt is used to grind the material. This abrasive belt is rotated by the single phase induction motor. In our project consist of end bearings with bearingcap, roller wheel, shaft, single phase induction motor and abrasive belt. This whole arrangement is fixed on the frame structure where the component rests. The roller wheel is mounted on the two end bearings with bearing cap by suitable arrangement. There are two roller wheel is used in our project to rotate the abrasive belt. One side of the roller wheel shaft, one v-pulley is coupled by the suitable arrangement. The single phase induction motor with V-pulley arrangement is used to rotate the abrasive belt through the belt drive mechanism. Belt grinding is an abrasive machining process used on metals and other materials. It is typically used as a finishing process in industry. A belt, coated in abrasive material, is run over the surface to be processed in order to remove material.

## 4.3 APPLICATIONS

- Belt grinding is a versatile process suitable for all kinds of different applications. There are three different applications of the belt grinding technology:
- Finishing: surface roughness, removal of micro burrs, cosmetic finishes, polishing.
- Deburring: Radiusing, burr removal, edge breaking.
- Stock removal: high stock removal, cleaning (e.g. of corrosion), eliminating mill or tool marks, dimensioning.

## 4.4 ADVANTAGES

- The machine is compact and rigid in size.
- Maintenance is less.
- It can be used on any place of small grinding application
- By varying the pulley diameter the speed of the abrasive belt to be changed.

## 4.5 DISADVANTAGES

- The abrasive belt should be changeable one for different material. This process takes more time.

## 4.6 GRINDING

BAINPOL VTD works to grind and polish finer abrasive particles until the desired surface quality is ready for chemical etching which by then the metallographic structure of specimen can be observed and measured with Vision engineering TIM5 or digital, optical, or electron microscopy.


**Figure 4.2 A sketch of the grinding machine, which is available in Government College of engineering, Bargur**

## 4.7 Automatic grinding method steps are:

1. Symmetrically load three to six mounted specimens into the specimen holder of an automatic grinding-polishing machine, with the flat surface of the ceramic section downward. Most manufacturers provide a leveling tool for loading the mounts into the holder. Attach the holder to the polishing head.

2. Grind the specimens at a contact pressure of 40 to 150 kPa on a bonded diamond platen for approximately 60 s or until the exposed surface of each specimen is flat and clean. Note that the pressure indicated on the grinding machine is usually the incoming air pressure, which is not necessarily equal to the pressure of the specimens against the platen. Perforated or grooved platens are available that aid in the removal . Experiment with the abrasive size, contact pressure, relative rotation directions (same or opposite), and frequencies shown subsequently to attain the best results. Typical machine settings: Contact pressure or frequency that is too high could damage the specimens or machine and shorten the life of the polishing cloth in the polishing steps. Contact pressure or frequency that is too low slows the rate of stock removal and can prevent any significant abrasion at all.

3. Remove the specimen holder from the machine and clean the specimens, as in Subroutine 4.1, but do not remove the specimens from the holder until the last polishing step is complete. Once clean, return the specimen holder to the machine for polishing or more grinding in successive steps on ever-finer abrasives and follow each step with thorough cleaning.

## 4.8 Manual Grinding:

The manual method is useful when automatic equipment is not available or when the depth of grinding is critical. Cross sections of microelectronic devices, such as multiplayer packages, often must be ground to a specific depth. To grind a Ceramographic section manually, choose a reference point on the specimen, such as point Q in the 12 o'clock position shown in Fig. 4.2(a). Hold the specimen surface firmly against the abrasive disc or belt such that the reference point is fixed with respect to the direction of abrasive motion. Continue grinding until the saw marks are replaced by Grinding and Polishing / 39 © 2002 ASM International. All Rights Reserved. Cerography: Preparation and Analysis of Ceramic Microstructures (#06958G)www.asminternational.org the parallel scratches of the first abrasive, as in Fig. 4.2(b). Clean the ground surface as described in Subroutine 4.1. Rotate the reference point Q to the 3 o'clock position, as in Fig. 4.2(c), and grind the specimen on the next finer abrasive until the previous artifacts are removed. The new parallel scratches lie at a 90° angle to the previous ones, as in Fig. 4.2(d). Rotation of the mount by 90° after each abrasive step (Fig. 4.2e) allows one to easily see when the artifacts of the previous preparation step have been removed. Clean the mount thoroughly after each step, as in Subroutine 4.1, to prevent transfer of abrasive particles from one platen to the next. In many cases, all the grinding can be accomplished in a single step.

## 4.9 POLISHING:

A machine which operates a rubbing-surface for bringing to a polish the surfaces of materials or articles to which a polish is desired to be given, as in polishing metals, stone, glass, wood, horn, or articles made from these or Other materials.


**Figure 4.3 A sketch of the polishing machine, which is available in Government College of engineering, Bargur**

## 4.10 Automatic Polishing:

After the finest grinding step, polish the specimens on nap less polishing cloths loaded with lubricant and progressively smaller diamond abrasives. Diamond polishing abrasives are typically available in 30, 15, 9, 6, 3, 1, and 0.25 µm sizes, in liquid suspensions, pastes, and aerosols. The suspensions can be automatically sprayed by some machines at timed intervals. Not every diamond size available is used or necessary in the procedure in Table 4.1. The transition from grinding to polishing may require additional time on the coarse polishing step to remove the artifacts of grinding. If paste is used, reapply it to the polishing cloth every few minutes. All types of diamond abrasives break down quickly and should be replenished frequently. Follow each polishing step with a thorough cleaning, as in Subroutine 4.1. Use nap less cloth for diamond pastes or suspensions and napped cloth for the alumina slurry or colloidal silica. Nap less cloth is a stiff, nonwoven PVC chemo textile sold under such trade names as texmet, Pellon, DP-Plan, MD-

Plan, and Pan-W. Now even, Fibre-reinforced-resin perforated pads and woven silk also work well for polishing ceramics with diamond pastes and suspensions. Flocked twill or napped cloth has a fuzzy texture that conforms to the surface being polished. Spread diamond paste, if used, on the cloth with a clean, gloved finger, along with additional lubricant. Polishing lubricants come under various names, including lapping oil, diamond extender, and blue lubricant. Be careful not to contaminate the paper with larger-size abrasive particles. Replace torn cloths immediately, being careful to smooth out any wrinkles or bubbles in the new cloth. Use xylene to dissolve the adhesive when removing worn-out cloth from the platen. Wear rubber gloves when using xylene. A worn-out cloth is easier to remove if the platen is first warmed with a heat gun. Platens tend to heat up during polishing and may require air cooling between intervals in order to prevent the polishing cloth from peeling or rupturing. Step 5(a) in Table 4.1, relief polishing, is optional. Relief polishing is not recommended when the specimen is to be tested for micro hardness. When edge retention is critical, such as on thin plates; or when the specimen will be viewed in high magnification, such as fine-grained microstructures. Relief polishing in conjunction with Nomarski differential interference contrast (see Chapter 7) can enhance the contrast

at low magnification by means of differential abrasion rates between harder and softer phases, for example, $Al_2O_3$ and inter granular glass in 85 to 98% alumina compositions, SiC and silicon in reaction-bonded silicon carbide, and between adjacent grains of $MgAl_2O_4$ spinel. Relief polishing can also polish the metal components in cross sections of microelectronic devices. Vibratory polishing with colloidal silica or alumina slurry, step 5(b) in Table 4.1, is another final polish technique. Each mounted specimen is clamped into a heavy brass or stainless steel cuplike holder. The weighted mount glides freely around a damp, napped polishing cloth on a vibrating platen for hours at atime. This method works very well for soft metals and semiconductors and is useful for some harder metals and ceramics. Ceramics that have low abrasion resistance and are not easily polished, such as AlN oftentimes, may be adequately polished by vibration on colloidal silica for 8 h. The colloidal silica suspension should be replenished every hour or so, a few millilitres at a time, and the napped cloth must remain damp. In somecases, a corrosive liquid is used along with the relief polishing slurry in a technique called attack polish. Attack polish combines mild etching and final polishing into a single step. Colloidal silica is suspended in a caustic solution that has an attack-polish effect on some materials. Either colloidal silica or a 1 to 10 mixture of Murakami's solution (see Table 5.1) to

0.05 μm γ-Al2O3 is recommended for the final polishing step on alumina with an abundant glass phase (Ref. 1). Murakami's solution is 3 g KOH and30 g K3 Fe (CN)6 in 60 mL distilled water. The attack polishing slurry is applied to chemically resistant synthetic fibre cloth rotating at 120 rpm for 30 min. The load is 15 N per 31.8 mm (1.25 in.) mounted specimen.

## 4.11 Manual Polishing:

After the finest grinding step and subsequent cleaning, manually polish the specimen onnap less polishing cloths loaded with lubricant and 15, 6, and 1 μm diamond paste, respectively. Rotate the specimen 90°, as in Fig. 4.2(a–e), and clean it thoroughly, as in Subroutine 4.1, after each polishing step. The relief polishing step with 0.05 μm γ- Al2O3 suspension is optional.

## 4.12 Coating characterization

Using a scanning electron microscope, the microstructure of the Al coating surface at random points and its cross-section were examined (SEM model JEOL JSM-6360LV). Samples were divided into sections, mounted in epoxy (Buehler trans-optic powder), and ground at 1200 grit. To guarantee that it was appropriate for metallography examination, polishing was then carried out using a 3-micron diamond suspension on Spec-Cloth. This was done in order to precisely assess coating thickness and have a better understanding of the distribution of porosity and splat formation. In order to identify the presence of cracking, voids, and phases in the coatings, coating cross sections were used. Additionally, energy-dispersive spectroscopy (EDS) was employed to ascertain the coatings' elemental makeup. The mean porosity and coating thickness were analysed and calculated using image analysis software (Image v. 1.47) and an optical microscope (ASTM E2109-01) [22–23].

According to ASTM E384-17, Vickers indentation was carried out on the coating's surface and cross section at random sites with a 300-gram load for 15 seconds[21]. To increase accuracy, ten indentations were produced. Under an optical microscope, each indent was examined to determine its legitimacy.

A profilometer (SJ-210Mitutoyo) with 0.05micron resolution and a 10micron maximum measuring range was used to determine the surface roughness (Ra).

To reduce mistakes, ten separate measurements of each sample were. It is important to note that the examination of coating qualities may have been impacted by undesirable phases at the location chosen for all testing.

As a result, testing for precision and consistency was done

# CHAPTER 5

## EXPERIMENTAL WORK

## 5.1 Development of LZ powder and its characterization

### Ball Milling Machine


**Fig 5.1 ball milling machine**

| | |
|---|---|
| Vial material | Porcelain Jar |
| Vial capacity | Porcelain Jar |
| Grinding media | Zirconia balls |
| Grinding media size | 10mm & 20mm dia |
| Max. Loading Capacity | 300g (Depends upon Density of material) |
| Min. Loading Capacity | 50g |

**Table 5.1 ball milling parameters**

A ball milling machine is a piece of equipment used for grinding and mixing materials, typically in the field of mineral processing, ceramics, and pharmaceuticals. The machine consists of a hollow cylindrical shell that rotates around its axis, partially filled with balls made of various materials such as steel, ceramic, or rubber. The balls act as grinding media, reducing the size of the materials being processed as they collide with each other and with the inner surface of the shell.

The shell is typically made of steel or other durable materials, and it is lined with wear-resistant materials to protect it from damage during the grinding process.

The balls: The balls used in a ball milling machine come in different sizes and materials, and their selection depends on the specific application. They are typically made of steel, ceramic, or rubber, and their size can range from a few millimeters to several centimeters in diameter.

The motor: The motor drives the shell to rotate at a controlled speed, which can range from a few revolutions per minute to several hundred revolutions per minute.

The control system: The control system includes sensors, actuators, and software that monitor and control the speed, direction, and other parameters of the milling process.

The loading and unloading system: The machine is typically equipped with a loading and unloading system that allows materials to be added and removed from the shell while it is in operation.

## 5.2 Zirconia balls


**Fig 5.2 zirconia balls**

Zirconia balls are ceramic balls made of zirconium dioxide (ZrO2), a high-performance material known for its exceptional hardness, strength, and wear resistance. Zirconia balls are often used as grinding media in ball milling machines for the processing of various materials, including minerals, pigments, and ceramics.

High hardness: Zirconia balls are extremely hard, with a vickers hardness of around 8.5, which makes them suitable for grinding and milling applications that require high wear resistance.

High strength: Zirconia balls are also very strong and can withstand high impact forces without cracking or breaking, which makes them ideal for use in high- energy ball milling.

Low wear rate: Zirconia balls have a low wear rate, meaning that they last longer and require less frequent replacement than other grinding media

## 5.3 Lanthanum oxide powder

- The mixture of $La_2O_3$ and $ZrO_2$ in the mole ratio of 1:2 weight percentage 56.8% and 42.96% respectively
- The mixture was milled for 4 h with (zirconia balls) ball mill media using single vial planetary mill TC Jar (250ml), Grinding Media-zirconia balls (10 mm dia - 20 Nos) at 300 rpm available at VB Ceramics, Chennai.
- The weight ratio of ball to powder was maintained at 10:1 and volume of the jar was around 250 ml.
- The milled powder was collected after 4 hours.

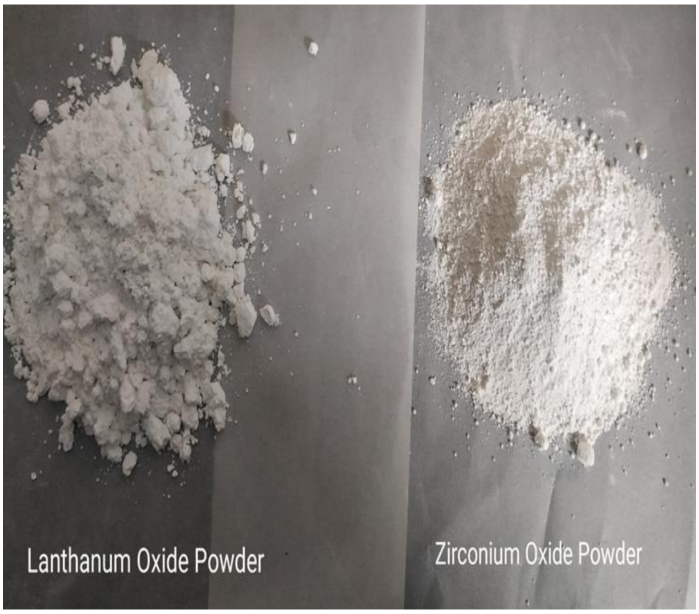


**Fig 5.3 Lanthanum oxide and zirconium powder**

## 5.4 Hydraulic pellet Press


**Fig 5.4 Hydraulic pellet Press**

A hydraulic pellet press is a machine used to compress powders or other raw materials into pellets of a specific size and shape. The press uses hydraulic pressure to apply force to the material being compressed, which causes it to form a pellet.

**Here are some components and features of a typical hydraulic pellet press:**

**Hydraulic system:** The hydraulic system is the main component of the press, and it consists of a hydraulic cylinder, a hydraulic pump, and control valves. The hydraulic cylinder generates the force needed to compress the material, while the hydraulic pump provides the pressure.

**Pellet die:** The pellet die is the component that shapes the material into the desired size and shape. It is typically made of stainless steel or other durable materials and is available in various sizes and shapes to accommodate different materials and applications.

**Pellet press chamber:** The pellet press chamber is the space where the material is loaded and compressed. It is usually made of stainless steel and has a piston that applies force to the material.

**Control panel:** The control panel contains the switches, buttons, and other controls that are used to operate the press, including the pressure and temperature settings.

**Safety features:** Hydraulic pellet presses are equipped with safety features to prevent accidents, such as emergency stops, safety guards, and interlocks.

## 5.5 LZ Pellet


**Fig 5.5 LZ PELLET**

- The milled powder was collected after 4 hours**.**
- Hydraulic pellet Press is used to compact Ceramic Powders into Pellet shape.
- The milled powders were then pelletized utilizing 25 mm diameter and 5 mm thickness by applying a 5-ton load with a dwell time of 5 min.


**FIG 5.6 LZ PELLET AFTER SINTERIG**

- The milled powder was sintered at temperatures of 1450°C for 4 h in air condition using a Box furnace.
- Then the sintered pellet was crushed in Ceramic crucible and sieved in different size of mesh.

## 5.6 Identification of Process Parameters

The choice of the spray parameters that will be investigated is the first stage. In general, all variables have an influence on the deposit's properties. Several trial experiments were conducted to determine the operational range of each parameter whileother factors remained constant. Based on earlier published research, the main criteria that had the greatest influence on the deposit were selected. The following qualities of ideal coatings were taken into account: suitable particle melting, flattening, and splat generation, all of which are highly dependent on process variables including power, SOD, and atomizing gas pressure. To determine the potential TWAS working ranges, several experimental investigations were carried out.

| | |
|---|---|
| Power (P) | 30 KW |
| Velocity (V) | 800 M/S |
| Thickness Of Coating | 350µm |
| Bond Layer Thickness | 150µm |
| Temperature | 15000K |
| Feed Rotation (Rpm) | 300 |
| Feed Rate (G/Min) | 30 |
| Standoff Distance (Mm) | 117 |

**Table 5.1 Coating parameters**

It would be costly and time-consuming to run trials with every potential operational parameter combination due to the high number of most important parameters. Therefore, it is essential to limit the number of tests needed to research the independent and effects of operational factors. The response surface technique

(RSM) was applied with a centrally composite rotating three-factor and five-level study was used with SOD, arc current, and primary gas pressure as inputs. The data was gathered in a random order. In order to prevent bias brought on by repeated settings, all procedure,equipment was reset between every spray cycle. Torch power was varied by changing the voltage on the control unit. As the wires broke off throughout the spraying procedure, the distance between them changed frequently, which caused the current to vary. Five power regimes were therefore taken for the studies, including low and high currents with an average of 50 to 250 A that generated 2.5 to 7.5 KW of power, respectively. The atomizing gas pressure was adjusted between 2 and 5 bars, and the standoff distance between the torch and substrate was fixed at 100 to 300 mm. The parameters used in this study are summarised in Table 5.1, along with the conditions under which they were processed.

# CHAPTER 6

## Result And Discussion

### 6.1 Hardness result

The Porosity-Hardness relationship in composite coatings as demonstrated in Fig 6.1 ,6.2,there is a relation between porosity and hardness. The hardness and porosity of the composite coating were based on the experimentation records derived. The experimental values are closely matched with a straight line. The regressive equation is utilized to represent the straight line.

Hardness (HV) = 1116.3 - 4.352 porosity (Vol. %)

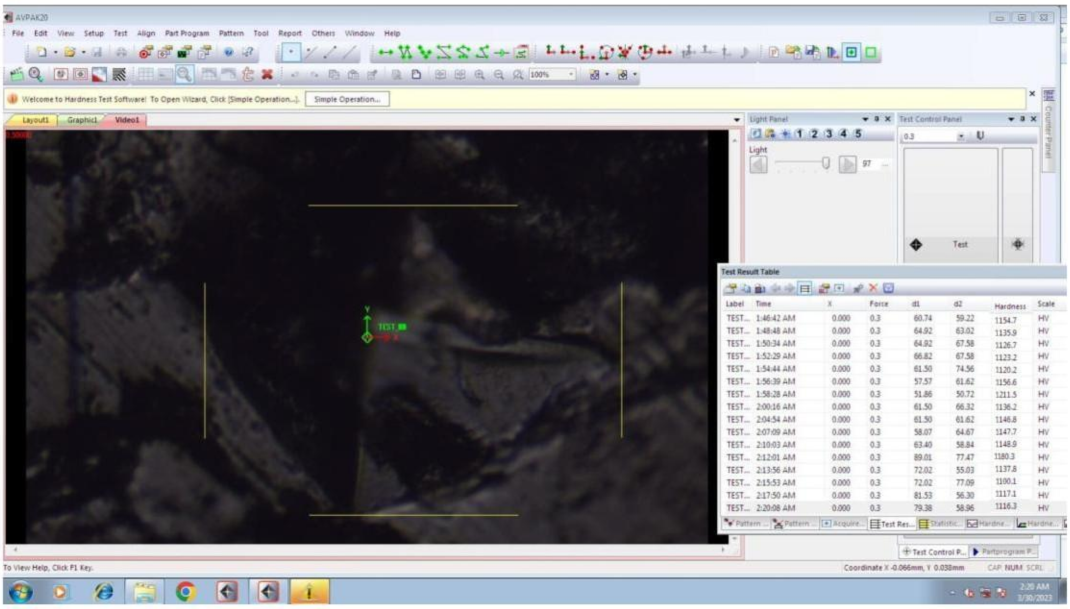


**Fig 6.1 hardness result**

- Inconel 718 hardness 610 HV
- After coating on Inconel 718 material hardness value is 1116.3 HV

## 6.2 POROSITY RESULTS

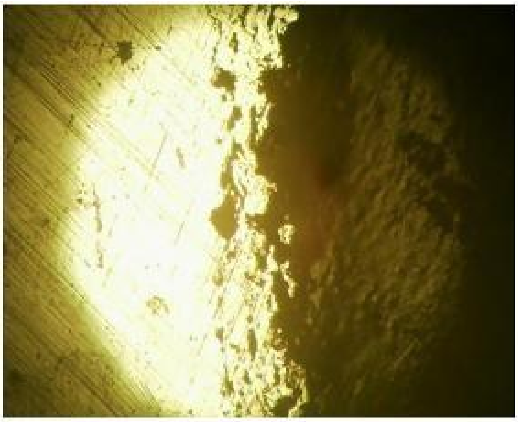

**Fig 6.2 porosity**

| FIELD | TOTAL OBJECTS | PORES% |
|---|---|---|
| 1 | 206 | 4.357 |
| 2 | 234 | 6.745 |

**Table 6.1 porosity result**

# CHAPTER 7

## 7.1 CONCLUSION

Plasma arc thermal barrier coating structures and process performance can be significantly altered by varying standoff distance, arc current, and atomizing gas pressures. Process associations were investigated using an ANOVA with a design of experiments approach with the intent of determining which spray parameters affect each of the coating properties. The data presented herein can be used as a guide when selecting operating conditions for a specific thermal spray application. , surface roughness, coating porosity, and spray particle size have all been analysed based on the variation of process inputs. In addition, a discussion of response surface shape and distribution tightness is included to further assist with input parameter selection. Coating deposition was most dependent on arc current. A high coating deposition was achieved with a combination of high primary gas pressure and high arc current.

- The initial step in the design of the experiment is to select the spray parameters that will be explored. Which are heavily dependent on process factors, namely power, standoff distance, and atomizing gas pressure.

- Identified the possible operating ranges for TPC process parameter with respect to power, standoff distance, and atomizing gas pressure.

- Empirical relations were established to predict (95% confidence level) the porosity¬ and hardness of LZ/YSZ coatings incorporating PSTPC sprayed variables including input power, standoff distance, and gas pressure.

- The developed model was evaluated using the ANOVA technique, with the desired¬ degree of confidence set at 95 %. The significance of the established associations was tested using the value and their respective probability values.

- The porosity and micro hardness of the LZ/YSZ coatings have been incorporated into a regression equation. If the porosity of the coating is specified, this equation could be utilized to estimate the hardness of Plasma arc sprayed coatings.

- Optimizing the TPC spray parameters were done using response surface methodology (RSM), which provides the minimum porosity and maximum hardness.

- As a result, the arc power has the greatest impact on the coating properties among the three variables, and the standoff distance and gas pressure are the subsequent important spray parameters.

- The input power of 30 kW, the standoff distance of 117 mm, and the temperature 15000 K of were the optimum deposition parameters for minimum porosity and maximum hardness of Plasma arc sprayed composite coatings.

# CHAPTER 8

## 8.1 REFERENCES


1. A.P. Newbery and P.S. Grant, Oxidation During Electric Arc Spray Forming of Steel, J. Mater. Process. Technol., 2006, 178(1–3), p 259–269

2. Arizmendi-Morquecho, Ana, et al. "Microstructural characterization and wear properties of Fe-based amorphous-crystalline coating deposited by twin wire arc spraying." Advances in materials science and engineering 2014 (2014).

3. B. Wu, Z. Pan, D. Ding, D. Cuiuri, H. Li, J. Xu and J. Norrish, A Review of the Wire Arc Additive Manufacturing of Metals: Properties, Defects and Quality Improvement, J. Manuf. Process., 2018, 35, p 127–139.

4. Chen, Y.,et al., Process optimization of a hybrid twin-wire arc sprayed composite coating Hanjie Xebio/Transactions of the China Welding Institution Volume 40, Issue 2, Pages 38 - 4125 ,2019.

5. D.L. Hale, W.D. Swank, and D.C. Haggard, In-Flight Particle Measurements of Twin Wire Electric Arc Sprayed Aluminum, J. Therm. Spray Technol., 1998, 7(1), p 58-63

6. DePalma, K., Walluk, M., Martin, L. P., & Sisak, K. (2022). Investigation of Mechanical Properties of Twin Wire Arc Repair of Cast Iron Components. Journal of Thermal Spray Technology, 1-14.

7. Gerashi, E., Alizadeh, R., & Langdon, T. G. (2021). Effect of crystallographic texture and twinning on the corrosion behavior of Mg alloys: A review. Journal of Magnesium and Alloys.

8. Horner, Allison Lynne, Aaron Christopher Hall, and James Francis McCloskey. "The effect of process parameters on twin wire arc spray pattern shape." Coatings 5.2 (2015): 115-123.

9. Ismail, N. S., Bakar, T. A. A., & Fadil, N. A. (2022). Effect of stand-off distance on the microstructural and mechanical properties of alzn pseudo- alloy coating prepared via wire arc spray process. Malaysian Journal of Microscopy, 18(1).

10. J. E. Gray and B. Luan, "Protective coatings on magnesium and its alloys -- a critical review," Journal of alloys and compounds, vol. 336, no. 1-2, pp. 88–113, 2002.

11. Jayakrishna, K., Kar, V. R., Sultan, M. T., & Rajesh, M. (2018). Materials selection for aerospace components. In Sustainable composites for aerospace applications (pp. 1-18). Woodhead Publishing.

12. Johnston, A. L., Hall, A. C., & McCloskey, J. F. (2013). Effect of process inputs on coating properties in the twin-wire arc zinc process. Journal of thermal spray technology, 22(6), 856-863. 46

13. K. Murugan, A. Ragupathy, V. Balasubramanian, K. Sridhar, Optimizing HVOF spray process parameters to attain minimum porosity and maximum hardness in WC-10Co-4Cr coatings, Surf. Coat.Technol. 247 (2014) 90–102, https://doi.org/ 10.1016/j.surfcoat.2014.03.022.

14. Karthikeyan S, Balasubramanian V and Rajendran R 2014 Developing empirical relationships to estimate porosity and microhardness of plasma- sprayed YSZ coatings Ceram. Int. 40 3171-3183