

Thermal Shock Resistant And Thermal Cyclic Fatigue Of Plasma Sprayed Thermal Barrier Samarium Zirconate Coatings

Submitted in partial fulfilment of the requirements for the completion of

PROJECT REPORT

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **SANJAY SHARMA.R (39260030), SANJAY.V (39260031)**, who carried out the project entitled "**THERMAL SHOCK RESISTANT AND THERMAL CYCLIC FATIGUE OF PLASMA SPRAYED THERMAL BARRIER COATINGS**" under our supervision from June 2022 to April 2023.

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ABSTRACT

In Thermal Barrier Coatings, composite top coat materials with high temperature stability and low thermal conductivity are greatly desired. Thermal Barrier Coatings (TBCs) with a two-layered structure consisting of an oxidation protective bond coat and a thermally insulating Yttria stabilised Zirconia (YSZ). This top coat are used in gas turbine engines and are used in aircraft propulsion system to provide thermal insulation and resistance from Oxidation to metallic components from the hot stream of gas. As a base metal, Hastalloy X has strong oxidation and corrosion resistance, and a bond coating of NiCrAlY was sprayed by plasma spray technique. Since their introduction in gas turbine engine components, plasma sprayed thermal barrier coatings have piqued the curiosity of researchers. Despite the fact that various new materials are being developed, YSZ remains the industry standard.

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LIST OF SYMBOLS AND ABBEREVIATIONS

ABBREVIATIONS	DESCRIPTIONS
TBC	Thermal Barrier Coating
YSZ	Yttrium Stabilized Zirconia
NiCrAlY	Nickel Chromium Aluminium Yttrium
TEC	Thermal Expansion Coefficient
APS	Atmospheric Plasma Spray
XRD	X-Ray Diffraction
SEM	Scanning Electron Microscopy
TGO	Themally Grown Oxide
TCF	Thermal Cyclic Fatigue
TSR	Thermal Shocking Resistance
OEM	Original Engine Manufacturer
EDS	Energy Dispersive X-Ray Spectroscopy
GPa	Giga Pascal
MPa	Mega Pascal

SYMBOLS	DESCRIPTION
K	Thermal conductivity
A	Thermal Expansion coefficients
wt%	Weight Percentage
°C	Celsius
°F	Fahrenheit
mm	Millimeter
LZ	Lanthanum Zirconate
CZ	Cerium Zirconate
ZrO ₂	Zirconium Oxide
Y ₂ O ₃	Yttrium Oxide
SZ	Samarium Zirconate
μm	Micrometers

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Thermal Barrier Coatings (TBCs) have the most intricate structures and perform in the most demanding high temperature conditions observed in aviation and commercial gas turbine engines. Coatings protect a variety of structural engineering components against corrosion, wear, and thermal insulation. Low thermal conductivity ceramic made of TBCs is used to protect metallic components from the hot gas stream in contemporary gas turbine engines used for aircraft and marine propulsion. Engines with thermal barrier coatings may run at greater gas temperatures when component heating is not as severe. This results in improved fuel control and longer component service life.

Hastalloy X, a nickel-based super alloy, was chosen because of its great strength at high temperatures. It is extremely strong and oxidation resistant up to 2200°F. Thermal Barrier spray coating is applied at high temperatures with varied proportions of different chemical compositions on the super alloy to improve the corrosion resistance capabilities of Hastalloy X nickel-based super alloy. Samarium zirconate and Yttria stabilised Zirconia are the chemicals employed in this procedure (YSZ).

Nickel-based super alloys have been chosen for aerospace materials in recent years due to their low density, superior increased temperature strength, and high melting point. Thermal barrier coating (TBC) systems and future gas turbine engine applications require the identification of coating materials that can function for prolonged periods of time and at greater surface temperatures. It is also vital to understand how the robustness of various bond-coats influences coating performance and longevity, as well as how they interact with the underlying super alloy.

1.2 GAS TURBINE

A Gas turbine is a type of heat engine that converts the energy released by fuel combustion into mechanical power that may be used to power a generator, an aeroplane, or other machinery. A gas turbine works on a simple principle: air is pulled into a compressor and compressed, then fuel is added and burnt in a combustion chamber, and the resulting high-temperature, high-pressure gas is expanded via a turbine, which drives the compressor and other gear.

Gas turbines are widely employed in power generation, aviation, and industrial applications. They have various benefits over other types of engines, including high power density, great efficiency, minimal emissions, and quick start-up and shutdown periods. Gas turbines can run on a range of fuels, including natural gas, diesel, and biofuels, making them a versatile and adaptable technology.

Gas turbines come in a variety of sizes and designs, ranging from tiny units used in power plants or oil rigs to massive ones used in aviation and industrial uses. They are also used in combined cycle power plants, where the gas turbine exhaust heat is utilised to produce steam, which is then used to drive a steam turbine, enhancing the plant's total efficiency.

Gas turbines have three main parts,

- Compressor
- Combustor
- Turbine

Compressor sucks in the air from outside of turbine and helps to increase its pressure. Combustor produce high velocity and high pressure gas by burning the fuel. Turbine extracts the energy from the gas coming out from the combustor.

The Main Types Of Gas Turbine Engines:

- Turboprop engines
- Turbojet engines
- Turbofan engines
- Turboshift engines

Gas turbine engines are majorly used to power aircraft, trains, ships, gas compressors, pumps and generators. Whereas marine and industrial gas turbines can run on diesel, natural gas and hydrogen.

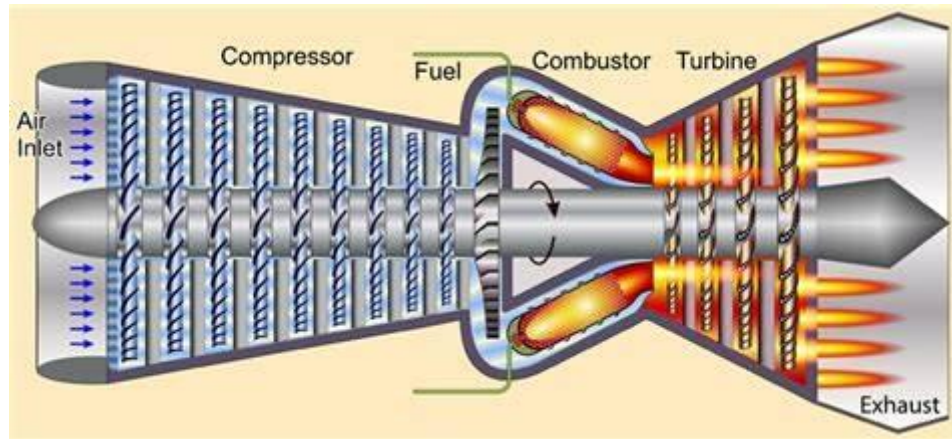


Fig1.1: A Gas Turbine Engine

1.3 SUPER ALLOYS

Super alloys are a type of material with great mechanical strength, corrosion resistance, and temperature stability. They are generally employed in high-temperature and high-stress situations such as gas turbines, jet engines, and nuclear reactors.

Super alloys are consists of metals like Nickel, Iron, Cobalt and Chromium, as well as additional elements like Titanium, Aluminium, and Tungsten. These metals were selected because of their capacity to produce a persistent oxide layer, which protects the alloy against corrosion and high-temperature oxidation.

These are metals that are far more heat resistant than conventional aircraft materials and can maintain its strength, stiffness and dimensional stability at greater temperatures. The properties of super alloys are

- ◆ High strength
- ◆ High corrosion resistance
- ◆ Machinability
- ◆ High thermal resistance
- ◆ Low coefficient of thermal expansion.

Superalloys can be divided into three groups: nickel-based, cobalt-based, and iron-based. Nickel based super alloys are often used in heated surroundings where there is a requirement of good mechanical strength, creep resistance and corrosion resistance . As a result, these type of alloys are what are to create modern power turbine discs for jet engines.

Super alloys are majorly used in Aerospace industries for Turbine blades and in Marine industry for Submarines and in Nuclear reactors and Industrial gas turbines. Some examples for super alloys are

- Hastalloy
- Inconel
- Waspaloy
- Rene alloys
- Incoloy

1.3.1 Nickel Based Super Alloys

Due to their exceptional mechanical and chemical rates, nickel- grounded super blends find expansive use in the chemical, nuclear reactor, aerospace, and marine sectors. One of the most remarkable parcels of nickel super blends that's utilised in spurt machines is their outstanding resistance in contrast to creep and stress rupture at high temperature. The top characteristics of nickel as an amalgamation base are its high phase stability of nickel matrix FCC and the capability to be strengthened by a variety of straight and circular means. Nickel – grounded super blends generally have 9 or further different essential ingredients that are directly controlled. utmost nickel – grounded blends contain 5 - 20wt chromium, upto 8wt Titanium and aluminium, 5- 10wt cobalt and small quantities of zirconium and carbon. Other additions that are commonly used to this admixture are molybdenum, tungsten, niobium etc. The most common type of nickel blends that substantially used in artificial as well as aeronautics operation are Hastalloy X, Waspalloy, IN- 718 and R100. The percentile compositions of separate blends have been colluded in the below table.

Table 1.1: Percentile Composition of Nickel Based Super Alloys

Alloys	Ni	Cr	Co	Mo	Nb	Ti	Al	Fe	C	B	Zr
Waspalloy	57	19.5	13.5	4.3	-	3	1.4	2	0.07	-	0.09
HastalloyX	47	22	1.5	9	0.5	0.15	0.5	18	0.1	0.008	-
INC-718	52	19	-	3	5.10	0.5	0.05	18.5	0.08	-	-
U720LI	57	16	15	3	-	2.5	2.5	-	0.02	0.01	0.03

1.3.2 Hastalloy X

Hastalloy X is a super alloy based on nickel that has high strength and oxidation resistance up to 2200°F. It has also demonstrated remarkable stress corrosion cracking resistance in petrochemical applications. This alloy's forming and welding characteristics are exceptional. Because of the convenience and economy with which Hastalloy X alloy can be manufactured, as well as its excellent tensile, fatigue, creep, and rupture strength, it is used in a wide range of applications. Because of its exceptional resistance to oxidising, reducing, and neutral atmospheres, it is recommended for use in industrial furnace applications. After 8,700 hours of operation at 2150°F, the furnace rollers of this alloy were still in good condition..

Aging process of this alloy will result in a increase in strength and hardness with the effect being related to hours of exposure at the aging temperature.

Table 1.2: Physical Properties of Hastalloy X

Properties	Metric
Density	8.220g/cm ³
Melting point	1316°C

Table 1.3: Mechanical properties of Hastalloy X

Properties	Metric
Tensile strength	755Mpa
Yield strength	385Mpa
Elastic modulus	205Gpa

Table 1.4: Thermal properties of Hastalloy

Properties	Metric
Thermal expansion co-efficient	13.0 $\mu\text{m}/\text{m}^\circ\text{C}$
Thermal conductivity	9.1W/mK

1.4 THERMAL BARRIER COATING (TBC)

Thermal Barrier Coating (TBC) enables current gas turbine engines to run at temperatures a little above the superalloy melting point (-1300°C), hence enhancing engine efficiency and performance. If the working temperature is somewhat lower, TBC helps decrease metal temperatures, making engine components last longer. The TBC is more sophisticated than any other coating system because to its multi-layer structure and demanding working conditions. TBC is the unique system in which complex interactions like as diffusion, oxidation, phase change, elastic deformation, creep deformation, thermal expansion, thermal conductivity, fracture, and sintering take place.

Advances in both the metal base and the ceramic top layer are being developed and implemented. Improved performance and durability, process advancements, and recognising and changing failure mechanisms are all examples of advancements. The turbine blades and vanes in the high temperature zone of the petrol turbine are made of a nickel-based super alloy with a high melting point. The current generation of TBC systems can withstand surface temperatures of up to 1275°C .

A TBC system consists of two layers. The first layer is a metallic layer or bond coat that is put on a nickel-based superalloy. The bond coat is a Ni-based alloy with several additional additives (such as Cr, Pt, Co, Y and Hf). The second layer is a top coat of Yttria Stabilised Zirconia (YSZ) placed atop the bond coat. The top coat plays a key function in thermal insulation and in the first layer where the bond coat protects the base alloy from oxidation through the production of second oxide.

Thermal conductivity and thermal expansion coefficient (TEC) are the most significant qualities to consider while selecting TBC materials. TBCs have an important role in resolving the problem of thermal stability in high-temperature engine components.

1.5 CERAMIC MATERIALS

A substance that's neither metallic nor natural is a ceramic. It might be either crystalline or glassy, or both. Ceramic accoutrements are principally a admixture of complexion, earthen rudiments, maquillages and water. Ceramic accoutrements are codified into manners grounded on their composition.

- **Silicate Ceramics** Accoutrements having composition of oxygen and silicon are called silicates. The main manners of silicate ceramics are grounded on either alumino silicates or magnesium silicates.
- **Oxide Ceramics** These are non-metallic and inorganic composites by nature that carry oxygen, carbon copy, or nitrogen. Oxide ceramics carry alumina, Zirconia, silica, aluminum silicate and other oxide accoutrements.
- **Non-oxide Ceramics** At veritably high temperatures and austere thermal shock conditions non-oxide ceramics has helped to beat extreme wear and tear and erosion cases. These pottery are exercised in medicinal, gas diligence, swirling corridor, break- plates, position legs for protuberance welding, abrasive greasepaint blast snoots and essence forming device.

- **Glass Ceramics** Glass ceramics are produced by a ruled crystalline procedure. They're principally poly-crystalline accoutrements. They partake numerous common or garden characteristics with both spectacles and ceramics.

1.6 COATING MATERIALS USED FOR HIGH TEMPERATURE MATERIALS

1.6.1 Yttria-Stabilized Zirconia (YSZ)

Yttria-Stabilized Zirconia (YSZ) is a ceramic that occurs when the inclusion of yttrium oxide stabilises the cubic crystal structure of ZrO_2 at room temperature. These oxides are also known as "Yttria" (Y_2O_3) and "Zirconia" (ZrO_2).

Zirconia (zirconium oxide) is a solid oxide that occurs in three various crystal forms depending on temperature: monoclinic, tetragonal, and cubic. The monoclinic-tetragonal transition is accompanied with a large volume change, which might be problematic when treating the material.

The material's cubic phase, which generally only develops at exceedingly elevated temperatures ($>2690^\circ\text{C}$), is stabilised during a wide temperature range by replacing some of the yttrium ions with zirconium ions. This makes the material a conductor of O^{2-} ions and enables the production of sintered Zirconia products. The greatest conductivity is reached at around 8 mol% Yttria and rises with temperature and yttrium dopant concentration.

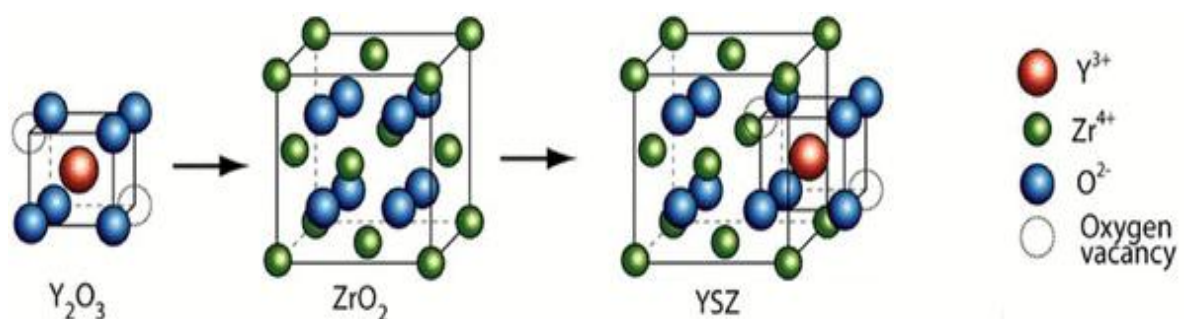


Fig1.2: Formation of YSZ

1.6.2 Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$)

Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is a ceramic substance from the rare earth metal oxide family. Its perovskite crystal structure and outstanding thermal stability, chemical durability, and radiation resistance make it a potential material for a variety of high-temperature applications such as thermal barrier coatings, solid oxide fuel cells, and nuclear waste immobilisation.

$\text{Sm}_2\text{Zr}_2\text{O}_7$ is also being researched for potential applications as a radiation detector and scintillation material in medical imaging devices. Because of its unique properties, it is a promising material for these applications because it can convert ionising radiation into light, allowing for radiation detection and measurement.

One of samarium zirconate's most significant features is its great thermal stability, which means it can resist high temperatures without deteriorating or dissolving. Its feature qualifies it for use in thermal barrier coatings, which protect high-temperature components from heat damage. It can be used to coat turbine blades in gas turbines, for example, to protect them from high-temperature gases.

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1.6.3 Lanthanum zirconate ($\text{La}_2\text{Zr}_2\text{O}_7$)

Lanthanum zirconate (LZ) is an excellent thermal barrier coating material since it exhibits lower thermal conductivity & greater thermal stability compared to various thermal barrier coating systems. In comparison to 8YSZ, LZ has many advantages for TBC applications, such as no phase transition from room temperature to melting temperature, high sintering resistance, a low coefficient of

thermal conductivity like 1.5 -1.8 W/m/K at 1273 K, and the primary benefit is LZ has lower ion diffusivity, which prevents oxidation of the bond coat and substrate.

The main drawback of LZ is that its CTE value is low in comparison to the bond coat and substrate CTE values, which do not match. The co-precipitation process, solid state reaction method, and sol-gel method are all used to make LZ powders. A water-based solution of lanthanum nitrate hexahydrate ($\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) and zirconium oxychloride ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$) with NH_3 solution is used to prepare LZ powders for the co-precipitation procedure. The LZ compounds are stable at ambient temperature, however at high temperatures, the compounds undergo a shift from pyrochlore into a defect fluorite structure. The LZ is deposited over the substrate by Atmospheric Plasma Spraying (APS) process. The APS coating specimens has lower thermal conductivity when compared with EB-PVD coated specimens.

The deposited LZ coating using SPS method and in the result of the test many cracks and pores were visible in the microstructure. Nano sized lanthanum zirconate was selected as topcoat material. It is confirmed that nanocomposite coating produced by the SPS process is suitable for protecting the substrate material from high temperatures. Lanthanum zirconate has mechanical properties like higher fracture toughness, higher hardness, lower young's modulus of rigidity. By the result of thermal cycling behaviour Double ceramic layer (DCL) based LZ thermal barrier coatings had greater residual stress, even the fracture resistance (KIC) is poorer than 8YSZ.

1.6.4 Neodymium Oxide (Nd_2O_3):

Thermal stress tests cause holes on the coating surface to form, which then expand into fractures and cause coating deterioration. They may be reduced by adding Nd_2O_3 particles, and the coatings became more compact, and the size of the micropores on the coating's surface increased while the number of micropores decreased. Additionally, when the concentration of Nd_2O_3 particles increased, the cracks shrank and eventually eliminated, suggesting that adding Nd_2O_3 particles to the electrolytes might significantly improve the coating's ability to withstand thermal shock. The coating hardness may be improved by adding Nd_2O_3 , and the

thickness of the coatings was initially raised and then decreased depending on the concentration of Nd₂O₃. As compared to the others, the modified Nd₂O₃/doped YSZ coatings exhibit better thermal cycle life and thermal shock resistance.

Exfoliation of the ceramic layer's inner layer is the most prevalent reason of thermal shock failure. As the rare earth oxide doping level rose, the luminous intensity of the coating raised and the Nd₂O₃/modified YSZ coating produces a broad spectrum after the thermal shock test lowers and shows the t' phase as the phase structure. Because of the high doping level of Nd₂O₃, the phase change of zirconolite-2M to zirconolite-4M increases as the concentration of Nd₂O₃ increases, the grain size of the crystalline phase reduces, and the synthesis of oxyapatite phase occurs, and the chemical durability increases.

The Nd₂O₃ layer formed by the low-cost sol-gel dip coating has strong insulating qualities that will be useful in future microelectronic applications. The SiO₂/Nd₂O₃ coating was laser ablation resistant, and it effectively protected the C/C substrates from oxidation and thermal deterioration. Thermal and chemical ablation were two types of laser-ablation damage that might be mitigated by enhancing the reflectivity of the coated surface. The SiO₂/increased Nd₂O₃coating's capacity to reflect light made it more effective in avoiding laser damage, greatly prolonging the service life of the repaired region, and successfully preventing secondary damage.

1.6.5 Cerium Zirconium Oxide (Ce₂Zr₂O₇)

Using a solution-assisted combustion synthesis method, crystalline cerium-zirconate (CZ) powders were created, and they were then calcined at 1123 K for 8 hours to create coarse crystalline powders. Studies of SEM (scanning electron microscopy) characterization were carried out to assess the morphology of the powders. The presence of crystalline CZ and a crystalline phase was confirmed by XRD examination of the resultant powder. Fluorite cubic lattice phase Ce₂Zr₂O₈ with disorder. Additionally, the atomic positions, lattice characteristics, and phase composition were examined. The XRD measurements were refined done to calculate the concentrations of CZ and Ce₂Zr₂O₈ phases.

Inconel 625, a Ni-base superalloy, was selected as the powders were applied using an air gun to coat the substrate spraying with plasma (APS). Thermally generated oxide (TGO) due to the high temperature of the Al_2O_3 layer that formed is the base. The use of coatings on superalloy substrates for a variety of intrinsic and extrinsic applications that were influenced by temperature is well known. Highly oxidising conditions demand protective coatings that don't spall easily.

Ceramic coatings with high wear and hardness resistance that is subjected to corrosive and heat conditions compared to metallic materials, they have a number of advantages due to their low densities. The heat conductivity of a ceramic top coat is significantly lower than that of a metallic one. It is feasible to create a substantial temperature by covering the components' internal surfaces and the substrate drop across the ceramic layer by a suitable amount (a few hundred K) choosing components for thermal barrier coatings (TBCs).

Years ago, Yttria Stabilized Zirconia (YSZ) was widely used in one of the most important ceramic topcoat materials [19]. Beginning with a solid solution of $\text{Ce}_{0.5}\text{Zr}_{0.5}\text{O}_2$, the pyrochlore CZ phase ($\text{pyrCe}_2\text{Zr}_2\text{O}_7$) with a high degree of cationic sublattice ordering is formed, and this phase is the precursor to the $\text{Ce}_2\text{Zr}_2\text{O}_8$ phase. The process of creating $\text{Ce}_2\text{Zr}_2\text{O}_8$ has reached its conclusion. phase contains a minor reoxidation of $\text{pyrCe}_2\text{Zr}_2\text{O}_7$ under air conditions at about 873 K conditions. However, the production of phase-pure $\text{Ce}_2\text{Zr}_2\text{O}_8$ (phase-pure and with the highest).

Given that the bulk of previous experiments used decreasing temperatures of 1573 K or less, The authors show that temperatures as high as 1300°C are required to enable phase-pure pyrolysis of CZ and $\text{Ce}_2\text{Zr}_2\text{O}_8$, but that temperatures as low as 1300°C are inadequate. A temperature of 1773 K is required to guarantee that both phases are pure. CeO_2 -based materials are employed in a variety of catalysis applications, such as a carrier for active metal particles, oxygen storage co-catalysts in vehicle exhaust gas post-treatment, and even as a catalytically engaged component. (without the use of noble metals), as in the Deacon reaction, which is a catalytic oxidation of HCl.

The primary responsibilities of Materials based on CeO_2 are used in automotive catalysts to balance the amount of oxygen in the exhaust gas by storing and releasing oxygen, flowing between the engine's lean and rich operation, respectively. The term "oxygen storage" appropriately describes this oxygen exchange of CeO_2 capacity".

1.7 COATING TECHNIQUES

The technique of creating a thermal barrier coating (TBC) involves applying molten or softened particles to impact onto a substrate. Modern TBCs are necessary to protect engine components from oxidation and hot corrosion in addition to limiting heat transmission through the coating. A temperature decrease can be generated over the layer's thickness by adhering a coating of a low thermal conductivity material to the surface of an internally cooled gas turbine blade. We need to note that at current operating temperatures to extend the lifetime of the component these types of coatings are used.

The earliest TBCs were aluminide coatings, used in the 1970s. In the 1980s, better ceramic coatings were accessible. These coatings improve the performance of turbine blades. Additionally, the coatings extend the life of the blades, in some cases nearly doubling it. The classification of spray techniques are Arc Wire thermal spray process, Detonation spray process, High Velocity Oxy Fuel process, Cold Spray coating process, Plasma spray process, ESVD, EB-PVD and SPPS. In this research we used plasma spray coating for the coating process. Among all above coating technologies, plasma spraying process is the most widely used technique for the coating purpose.

1.7.1 Atmospheric Plasma Spray Coating (APS)

The Plasma spray process involves scattering molten or hot sapped material applied to the face in order to produce a coating. A truly hot tube honey is used to snappily toast and accelerate cream, which is also fitted into the honey. The heated substance makes contact with the substrate's face and snappily cools to

produce a coating. Tube spray deposit is a process where it combines flyspeck melting, quenching and connection in a single operation.

In this process it involves to fit the material to be deposited in a tube spurt, where the material is melted and the molten patches strike the substrate face forming an convert coating. The paraphernalia can be fitted to the tube spurt in form of cream scattering or line spraying. The tube spray cannon is made up of a water-cooled bull anode and tungsten cathode. Tube gas (argon, nitrogen, hydrogen, and helium) travels around and through the anode, which is formed like a constricting nozzle. The heat resistance from the bow leads the gas to attain high temperatures, causing it to detach and ionise, resulting in the formation of plasma. Powder is often injected into the passageway honey via an exterior cream harborage positioned close the anode snoot outlet. The cream is quickly heated and accelerated, resulting in spray distances ranging from 25 to 150 mm. The main benefits of tube spraying are that it has a relatively low complexity setup and that a broader range of coating equipment and coating processes may be applied is fast.

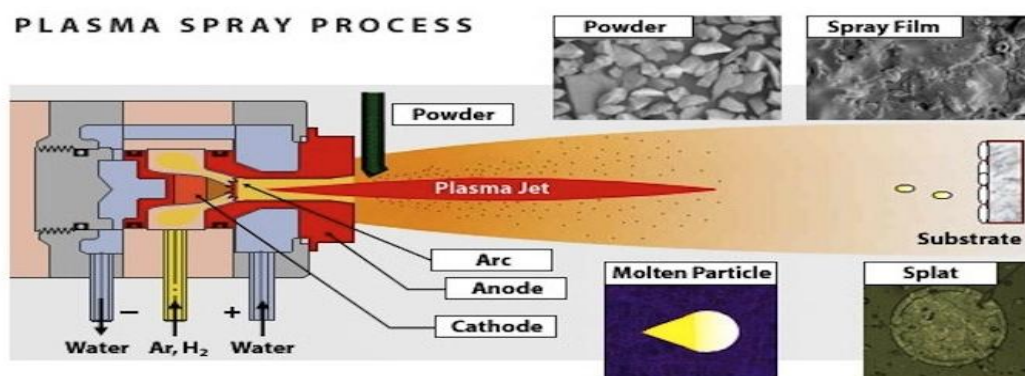


Fig1.3: Schematic diagram of Plasma spray process

1.7.2 Electron Beam Physical Vapor Deposition (EB-PVD)

An electron beam is used in the EB-PVD process to raise the temperature of a permeable object of the substance that is coated in a vacuum environment across the melting point to approximately 3500°C. Porosity is essential for the object to withstand the rapid heating caused by the electron beam. The coating is formed by condensation as the substrates spin or migrate in a planetary motion. Ceramic top coatings for TBCs made by the EB-PVD technique are made up of loosely bound, columnar-shaped particles and have an elevated level of mechanical compliance. Because there are no large droplet boundaries due to the heat flow direction, these coatings have a reasonably high thermal conductivity.

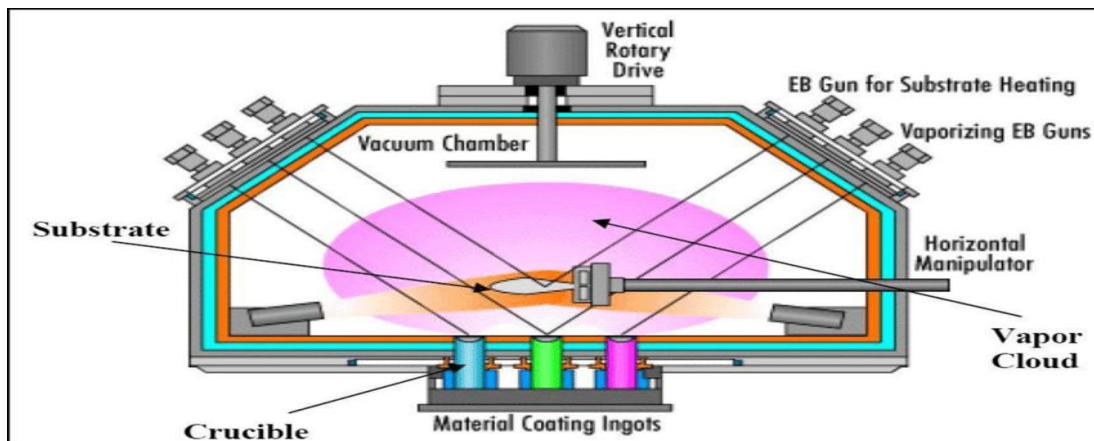


Fig1.4: Schematic diagram of EB-PVD process

1.7.3 High Velocity Oxygen Fuel Process (HVOF)

High Velocity Oxygen Fuel (HVOF) coating is a thermal spray spraying method used to enhance the surface of components. HVOF coating works by combining liquid gasoline and oxygen, that is then pumped into and fired in a combustion chamber. The resulting gas has exceedingly extreme pressure and temperature and will be expelled at supersonic speed by the nozzle. A jet of steaming gases and powder is aimed at the surface to be coated. The resulting thick covering has low porosity, high bond strength, and a variety of benefits, including corrosion resistance.

High Velocity Oxy-Fuel Process

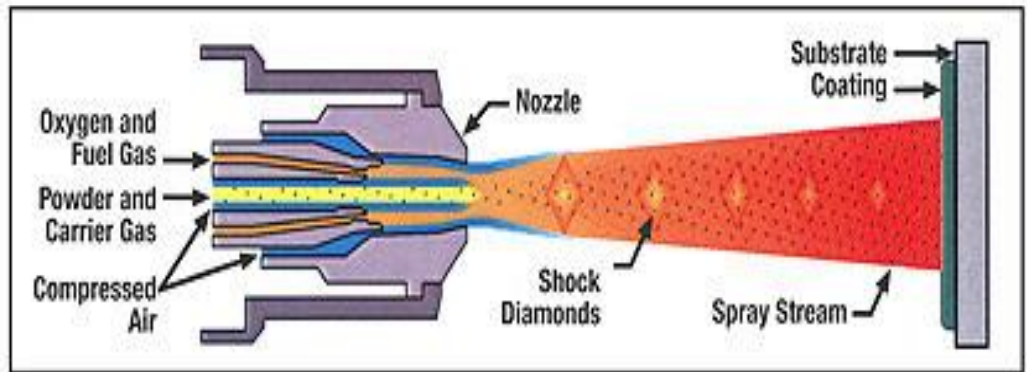


Fig1.5: Schematic diagram of HVOF process

Advantages of HVOF coating:

- Due to greater particle impact velocity, it has higher density and lower porosity.
- Due to harder and tougher coatings it shows better wear resistance.
- Thicker coating due to less residual stresses

Disadvantages of HVOF coatings:

- The attributes and microstructure of HVOF sprayed coatings are exceedingly complicated, with several processing factors influencing their properties and microstructure.
- Because of the necessity for tight size distributions, powder sizes are limited to a range of around 5 - 60 μ m.
- For this coating procedure to be safe and provide consistent coating quality, well-experienced and quality employees are necessary.

1.7.4 Electron Beam - Direct Vapor Deposition (EB - DVD)

The system makes use of a continuously operating 60 kV/10 kW radial e-beam cannon that has been modified to work in a low vacuum condition! with a variable-composition He-O₂ carrier gas jet. In this process, the vaporised

materials gets absorbed in a carrier gas jet and rapidly deposited onto the surface of the substrate, resulting in excellent material utilisation efficiency. The velocity and flux of atoms of gas entering the chamber, as well as the nozzle parameters and operating chamber pressure, may all be considerably altered, allowing for a wide range of handling conditions.

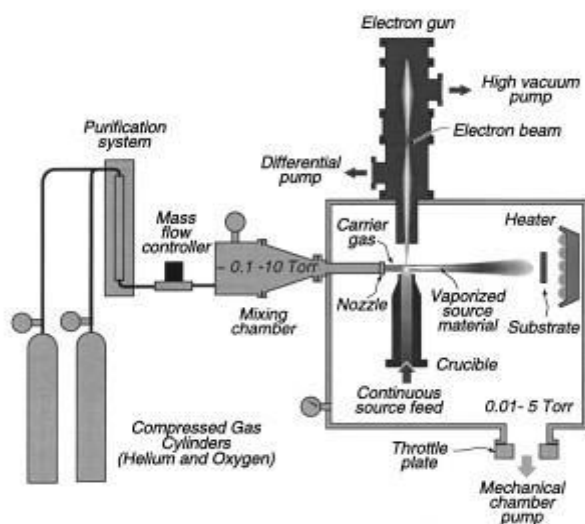


Fig1.6: Schematic diagram of DVD process

Direct vapor deposition is a chemical vapor deposition (CVD) process for producing solid thin films and coatings. CVD condenses vapor phase chemicals to produce solid phase material, which can alter the optical, electrical, mechanical, and corrosion resistance of many substances. Direct vapor deposition involves the vaporization of initiator materials and monomers, which are then injected into a vacuum chamber holding the material to be coated. Because of its low cost, high yield, and simplicity of scale-up, this process is frequently utilized in commercial production of carbon nanotubes.

The CVD process starts with tanks filled with an initiator substance and one or more monomers, which are the building blocks of the desired polymer covering. They are vaporized, either by heating or by lowering the pressure, and then delivered into a vacuum chamber holding the coating substance. As the monomers settle on the surface, they react to produce polymers. Direct vapor deposition is a flexible and commonly used manufacturing technology that can produce uniformly thick coatings even on complicated forms.

There are several commercial uses for direct vapor deposition. For example, researchers created a roll-to-roll processing method employing CVD to create sheets of graphene, a material with uses in electronics and other fields.

1.7.5 Detonation Method

Detonation coating is a thermal spraying method that produces a coating by heating and accelerating the coating material's powder feedstock with high-pressure, high-temperature gaseous products produced by controlled explosive combustion of a precisely calculated combination of gases. The detonation spraying coating is largely mechanically bonded, which is not as strong as metallurgical bonding.

The method is a "line of sight" procedure, which means that components must normally be coated before being used or assembled. Techniques like as optical microscopy, X-ray diffraction, and field emission scanning electron microscopy/energy-dispersive analysis have been used to study the coatings produced by detonation spraying.

1.8 METHODS OF COATING'S CHARACTERIZATION

Several procedures are used to completely describe the coatings. The most basic technique to control the coating's quality is to carefully examine its surface. Coating qualities affect how they behave in service. The best possible test is one conducted under simulated 'real-service' settings. Such a test, however, is costly, and sprayed coatings are often defined by examining their physical or chemical characteristics.

Overall, high-temperature coating characterization necessitates a mix of approaches that can offer information on the coating's morphology, composition, thermal stability, and mechanical characteristics. These approaches can aid in the identification of coating flaws or degradation processes, which is critical for assuring the long-term performance of coated materials in high-temperature settings.

1.9 HIGH TEMPERATURE CORROSION BEHAVIOUR:

High-temperature corrosion is a chemical reaction that happens when a metal comes into contact with hot gas at temperatures above 400°C in the absence of a liquid water phase. When diesel engines, gas turbines, equipment, and furnaces come into contact with hot gas containing specific pollutants, high-temperature corrosion can occur. High-temperature carburization, oxidation, and sulfidation are the most prevalent types of high-temperature corrosion. When the flue gas temperature exceeds 500°C, high-temperature corrosion can develop in super heaters and furnaces.

1.9.1 Thermal Cyclic Test:

A Thermal cycle is a procedure in which microencapsulated PCM (phase change material) is melted and then immediately frozen. PCM is a substance capable of absorbing and releasing significant quantities of heat energy while remaining at a steady temperature. The thermal and chemical behaviour of the material should not alter as the number of thermal cycles increases in the micro-encapsulated PCM application.

A thermodynamic cycle is a closed system in thermodynamics that experiences different changes due to temperature, pressure, and volume, but its final and starting states are equal. The capability of a fragile material to repel abrupt temperature changes is known as Thermal Shock Resistance (TSR), also known as thermal stress resistance. Without fail The TSR can be described as the maximum permitted temperature differential in the body and relies on the heating conditions (steady- state inflow, flash heating, heating by convection or radiation.). carpeted turbine blades are tested in a standard thermal cycle furnace test outfit.

Thermal barrier coatings (TBCs) are used in gas turbine and diesel engine uses to shield components from high-temperature environments. Thermal cycle fatigue (TCF) testing is used to assess TBC longevity and dependability. TCF testing includes exposing TBCs to sequential heating and chilling, which creates thermal stresses that can cause coating cracking and delamination. Several experiments have been conducted to examine TBC TCF behaviour.

1.9.2 Thermal Shock Test:

When there is a sudden shift in temperature, a phenomenon known as thermal shock takes place, causing a temporary mechanical load to be placed on an item. Due to the temperature shift, the object's various portions differentially expanded, which is what led to this load. A homogeneous material may be heated or cooled unevenly, leading to thermal shock, while a non-uniform material may be heated evenly.

Particularly in ceramics, it can cause the tensile tension to grow and fractures to spread. Using ceramics has a lot of problems because of thermal shock. As an illustration of thermal shock, consider the fracturing of ice in a hotter liquid and the uneven cooling of ore-bearing rocks next to a heat source.

Despite their well-known fragility, ceramics may be manufactured to tolerate thermal stress. Compressive stress can be added to a material to make it resistant to thermal shock, as is the case with tempered glass. Another strategy is to lower the material's Young's modulus. A material's ability to withstand thermal shock can be increased by increasing its toughness by fracture tip blunting or crack deflection, using the phases of plastic deformation and phase change.

Using ceramics, carbon/carbon, cement, and ceramic matrix composites might provide problems due to thermal shock. With more convective heat transfer, the sustainable temperature spike is reduced.

To shield components from hot surroundings, thermal barrier coatings (TBCs) are utilised. To assess the effectiveness of TBCs, thermal shock testing is done. Rapid temperature variations are used to simulate the product's life cycle during the test of the coating. A dual climate chamber is used for the test, moving the product quickly from one extreme of temperature to the other.

Manufacturers of industrial gas turbine engines and aeroplanes employ a variety of thermal shock testing techniques for both quality assurance and characterising new TBC systems during the development phase. In many aviation applications, the cyclic boiler oxidation test (FCT) is employed. The heat gradient interface are both significantly increased during the jet engine thermal shock (JETS) test. A hob rig test served as the basis for the JETS test.

CHAPTER 2

LITERATURE SURVEY

2.1 INTRODUCTION

Thermal barrier coatings (TBCs) are refractory-oxide ceramic coatings that are put to the surfaces of metallic components in the hottest areas of gas turbine engines. TBCs aid in lowering the temperature of the combustion chamber lining by 473°C, resulting in more effective gas-turbine engines. TBCs are manufactured to increase the effectiveness of hot section components such as turbine blades, vanes, and combustors. Thermal cycle fatigue testing is a critical step in determining the longevity of TBCs. The testing mimics the product's exposure to rapid temperature changes varying from -60°C to 170°C during its life cycle. The coating is tested for a significant number of rounds, and it is deemed damaged if it can no longer perform its heat protection function.

Several studies have been performed to study the thermal cycle fatigue behaviour of TBCs, and models to predict the life of TBCs during thermal cycling fatigue tests have been created.

2.2 LITERATURE OF THERMAL BARRIER COATINGS

Huang *et al* (2020) Thermal cycling behaviour was investigated as microstructural evolve, phases and fundamental analysis of substance piston crown material, aluminium oxide/Yttria Stabilised Zirconia (20%Al₂O₃ /80%8YSZ) and ceria/Yttria Stabilised Zirconia (20%CeO₂ /80%8YSZ) were coated across CoNiCrAlY bond coat on aluminium alloy (Al-13%Si) at 800 degree. The experimental results demonstrated spallation in Al₂O₃/8YSZ TBC owing to thermally generated oxide (TGO) and the buildup of residual stresses at the top and bond coatings, as well as an improvement in thermal efficiency and a reduction in particular consumption of fuel for both TBC coated engines.

Zhou *et al* (2020) Comparing the Thermal cyclic behaviour of Hf doped (Ni,Pt)Al bond coats with conventional (Ni,Pt)Al at 1100 °C to investigate the effect of addition of Hf into (Ni,Pt)Al. By the addition of Hf results in improved thermal cycling resistance, lower oxidation rate and decreased scale rumpling tendency and using thermal cycles is used to determine the Young's modulus, hardness in top coat. Due to oxidation and hafnium oxide formation the Hf diffuses from top coat while exposure to high temperature for long time.

Lashmi *et al* (2020) The TBC consist of EB-PVD technology-based deposition of GEZ/YSZ which exhibits the defect fluorite structure and here TBCs is the composition of GEZ coating, YSZ coating and NiCoCrAlYHf coating. Due to Er Stabilized $Gd_2Zr_2O_7$ and high TEC and low thermal conductivity in as deposited TBCs exhibit the high thermal shock life and thermal cycle life. The thermal cyclic test also revealed the TGO growth, TGO broken and crack evolution and formation of Ni-Al-Cr-Co-Y oxides. The spallation and delamination failure is due to the extension of crack which is caused by the diffusion of elements.

2.3 LITERATURE OF YTTRIA STABILIZED ZIRCONIA (YSZ)

Mohammadi, Poursaeidi, and Torkashvand (2019) To improve the durability of YSZ ceramic coating by tailored plasma sprayed, the strain tolerance and sintering resistance was developed and show thermal shock life as prolonged by more than four times. Due to rapid sintering induced stiffening during thermal expansion of top coat of conventional TBCs a premature spallation is occurs, whereas in new coated exhibits an enhanced sintering resistance which is used to preserving good strain tolerance. The interfacial cracking of TBC which is dependent on the stiffness of the YSZ which is resulted by the Cohesive zone finite element modelling and interfacial cracks may initiated due to the increase in elastic modulus acceleration and also results in large cracks. The rapid sintering during high temperature results the premature failure of conventional TBC and by decreasing the sintering-induced cracking the anti-sintering capacity and the durability of new tailored TBC is prolonged.

Gonzalez-Julian et al (2020) Here Inconel 625 is used as base metal in which Dense Ceria-Yttria-Stabilized Zirconia (D-CYSZ)/Yttria Stabilized Zirconia (YSZ) was deposited as bilayer TBC. The bilayer TBC shows excellent in thermal shock test in which exceeded 500 cycles with the delamination of coating below 20% and also exhibits the higher thermomechanical resistance than the conventional YSZ. The thermal shock resistance of TBC is improved when compared to conventional YSZ by the addition of LP-YSZ layer between Bond Coat and YSZ layer and resistance against stress is due to the increase of stiffness. The bilayer system shows less morphological and microstructural degradation and less infiltration of molten salts and better cohesion in YSZ without a dense layer and these proves that D-CYSZ layer is efficient barrier for molten salt attack

Feng *et al* (2020) Due to excellent thermophysical properties the $\text{Ba}(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{O}_3$ (BMT) ceramic is considered as TBC application, where the single layer BMT coating shows the low fracture toughness and poor chemical compatibility with thermal grown oxide. The performance of thermal shock BMT-YSZ coatings is better than the single layer BMT coating and the stress generated by $\text{Ba}_3\text{Ta}_5\text{O}_{15}$ phase and low fracture toughness of BMT causes the spallation of BMT-YSZ coating. No cracking and impurity phases and good deposition quality are exhibited by both plasma-sprayed BMT-YSZ and BMT/YSZ-YSZ composite coating. BMT/20 wt% YSZ-YSZ composite coatings have better thermal shock when compare to single-layer BMT coating, BMT-YSZ coatings and during thermal shock the BMT-YSZ coating exhibits layer-by-layer spallation which is due to stress generated and low fracture toughness of BMT. The fracture toughness of coating is improved and delay the formation of destructive cracks and shows better thermal shock performance by the addition of YSZ in BMT layer.

2.4 LITERATURE OF THERMAL CYCLIC TEST

Venkadesan and Muthusamy (2019) The three multilayer TBCs are investigated for thermal cyclic failure in which the TBC is based on $\text{LaMgAl}_{11}\text{O}_{19}$ (LaMA)/YSZ. It exhibits the good number of cycles like 11,749 cycles of its lifetime by optimizing the weight ratio and thickness of the intermediate LaMA/YSZ

composite layers and the failure of TBC is due to the formation of cracks due to sudden spallation around the YSZ layer and the spallation is not only occur at YSZ but also at interface of LaMA and adjacent intermediate gradient composite. The reduced cycling lifetime is due to the accelerated TGO growths during high temperature of bond coat which is caused by oxygen transportation facilitated by cracking mode and higher YSZ thickness also contribute for TGO growth rate.

Jiang *et al* (2020) To overcome the issues such as phase transition, thermal stability, thermal cycling life in single layer YSZ TBCs the bilayer/multilayer topcoat is used. Most promising among other materials GZO, LZO, LCO are the emerging materials for TBC, since these materials exhibits the factors such as fracture toughness, good sintering rate and even have better performance. Improved thermal cycling life and increased durability of TBCs are demonstrated by using the DCL TBCs and the increased durability, higher strain tolerance is exhibited in high rate by composite layer over single YSZ. The contemporary method or processing technique for the fabrication of multilayer TBC are SPS, SPPS and PS-PVD which also consist of both intrinsic and extrinsic failure modes.

Shen *et al* (2021) To compare its thermal cycling test with other materials, a double ceramic layer (DCL) thermal barrier coating is formed with high-entropy rare-earth zirconate (HE-REZ) as the topmost layer and Yttria Stabilised Zirconate (YSZ) as the inner layer. During the thermal cycle test, this HE-REZ/YSZ DCL coating showed elevated thermal durability, resistance to thermal shock due to the anti-sinterability of the materials, sluggish diffusion effect, and a greater match in the thermal expansion coefficient. When compared to the LS system during cycle tests, the LS system has a 53-times longer lifespan. The HE-REZ ceramic coating retains its fluorite structure due to high-entropy slow diffusion of cation and vacancies in oxygen during thermal cycling, while the $\text{La}_2\text{Zr}_2\text{O}_7$ coating undergoes phase change from fluorite to pyrochlore throughout thermal cycling.

Zhang *et al* (2019) Comparing the YSZ with $\text{Gd}_2\text{Zr}_2\text{O}_7$ and $\text{La}_2\text{Zr}_2\text{O}_7$ with respect to the thermal cycling resistance which in search of alternative materials for YSZ. The higher strength is exhibited by YSZ/ $\text{Gd}_2\text{Zr}_2\text{O}_7$ and $\text{La}_2\text{Zr}_2\text{O}_7$ /YSZ when compare to YSZ and the YSZ/ $\text{Gd}_2\text{Zr}_2\text{O}_7$ exhibits better performance whereas $\text{La}_2\text{Zr}_2\text{O}_7$ /YSZ exhibits lower performance during the cycling test. As better results

in both TGO growth and spallation, low elastic modulus and low thermal conductivity of both $\text{Gd}_2\text{Zr}_2\text{O}_7$ and $\text{La}_2\text{Zr}_2\text{O}_7$ show that double layered is best.

2.5 LITERATURE OF THERMAL SHOCK TEST

Huang *et al* (2020) Lanthanum-cerium-tantalum-oxide (LCT) solid solution is produced by adding Ta_2O_5 with $\text{La}_2\text{Ce}_2\text{O}_7$ (LC) which exhibits the high thermal stability between room temperature and 1573K (up to 1000 h). Since $\text{La}_2\text{Ce}_2\text{O}_7$ (LC) show higher phase stability, low thermal conductivity and large thermal expansion coefficient (TEC) than YSZ and as also shows sudden decrease in TEC at low temperature which is an drawback. These thermal cyclic test results that thermal shock resistance exhibited by LCT TBC is better than LC TBC and during cycling the spallation failure is occurred within the top coat and these affects the thermal lifetime which also due to TEC mismatch stresses, low fracture toughness and coating composition.

de la Roche *et al* (2020) For gas turbine application Mg_2SiO_4 is used as the TBC, since it shows excellent phase stability upto 1573K and 20% lower thermal conductivity than YSZ and its mechanical properties including hardness, fracture toughness, and Young's modulus is good than YSZ. As the water quenching test exhibits that thermal shock resistance of Mg_2SiO_4 is better than 8YSZ and the coating composition withstand upto 830 cycle at 1273K which is mainly relative to its microstructure evolution, excellent sintering resistance and higher fracture toughness. The coating recrystallization, thickening of interaction layer, thermal expansion mismatch results in the spallation and to improve the thermal cycling life by optimization of process and by using the double ceramic layer coating with YSZ.

Khan *et al* (2021) Gadolinium zirconate ($\text{Gd}_2\text{Zr}_2\text{O}_7$, GZO) should be considered for TBC due to its low thermal conductivity and better phase stability, sintering resistance, and calcium-magnesium alumino-silicate (CMAS) attack resistance at temperatures above 1200oC, as well as its low fracture resistance and formation of deleterious interphase with thermally grown aluminium. Thermal cycle tests demonstrate that both TBCs systems have a longer lifespan, a higher

strain tolerance microstructure, and superior sintering resistance than typical single-layered APS YSZ. A cold shock caused failure under CMAS in both multi-layered TBCs, which differed from failure in typical a single-layer APS YSZ TBCs. Under CMAS assault, triple layered TBCs have the longest lifespan, are more resistant to cold shock, and have greater CMAS infiltration resistance.

Cao, Ning, and Wang (2021) The investigation of thermal shock and thermal insulation properties of lanthanum magnesium hex aluminate ($\text{LaMgAl}_{11}\text{O}_{19}$)/Yttria stabilised Zirconia (YSZ) TBC and also compare with bilayer coating of $\text{LaMgAl}_{11}\text{O}_{19}$ and YSZ (DC-TBC). The improved thermal stability and high cycle lifetime when compare to as-sprayed DC-TBC is revealed by the thermal shock test at 1100°C and whereas in DC-TBC spalled at the interface which is due to the difference in the coefficient of thermal expansion between $\text{LaMgAl}_{11}\text{O}_{19}$ and YSZ layers and the cycle life time both coating is improved by the laser glazing of segmentation cracks. In both as-sprayed and laser-glazed condition the FG-TBC shows better thermal shock resistance, thermal insulation capability, higher back wall temperature and higher cycle lifetime compared to DC-TBC. The best heat management is exhibited bt the lager-glazed FG-TBC in terms if both thermal shock resistance and thermal insulation capability and it can also used in the application such as gas turbine.

Chen *et al* (2019) Forsterite-type Mg_2SiO_4 can be used for TBC application which possessed the good phase stability upto 1573K and exhibits 20% lower thermal conductivity at 1273K than 8YSZ and also prevents the coefficient of thermal expansion. The hardness and fracture toughness and Young's modulus of Mg_2SiO_4 is sightly lower than that of 8YSZ and shows low sintering activity. The durability of thermal cycling of Mg_2SiO_4 TBCs is better at 1273K and it can be improved by optimization of the parameters and by using the double layer ceramic coating with 8YSZ.

CHAPTER 3

AIM AND SCOPE

3.1 AIM AND SCOPE

The Aim and Scope of the project is clearly been described in this chapter. The project's objective is to carry out a comprehensive survey of the works released in the area of thermal cycle fatigue testing of thermal barrier coatings (TBCs) in order to find state-of-the-art developments and probable future research paths. Gathering information from scholarly sources, assessing the strengths and flaws of individual sources, and synthesising the information collected into an organised framework that supports the research question are all part of the project's scope.

3.1.1 Aim Of The Project

In this work phase stability and oxidation stability and thermal shock and cycling lifetime of Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) mixed with YSZ coated on the Hastalloy X which is an nickel based super alloy is studied.

3.1.2 Scope Of The Project

The investigation on the Atmospheric plasma spray coating of Samarium zirconate added Ytria Stabilised Zirconia has given insight into the effect of Samarium on the thermal cyclic and thermal shock conditions. Further investigations on the properties of Samarium zirconate added plasma spray coating and its behaviour at various conditions to understand the behaviour of Samarium added plasma spray coatings. Therefore, based on the investigation inclusion of Samarium zirconate on YSZ gives higher phase stability, oxidation stability and good thermal cycling resistance and thermal shock resistance.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS AND METHODS

The Materials and Methods part is critical in giving a comprehensive description of the study's methods and techniques. This part contains a thorough description of the materials used, as well as any equipment, tools, or chemicals. The methods part explains how the study was carried out, including the study plan, data gathering methods, and data analysis techniques.

4.2 INTRODUCTION

Hastalloy X is a nickel-based superalloy which consist of expansive use in the chemical, nuclear reactor, aerospace, and marine sectors. Hastalloy X exhibits excellent resistance to oxidation and corrosion at elevated temperatures. It has a solid solution strengthened, offering outstanding strength at high temperatures and stress rupture characteristics above 790°C or 1450°F. It is suitable for applications at temperature limits about 1200°C or 2150° F. The corrosion and oxidation resistance of alloy may decrease due to the high temperature involved in gas turbine operations. It can be overcome by depositing the Ytria Stabilised Zirconia on the Hastalloy X which will acts as the thermal barrier coating and gives protection to the materials. But it also exhibits the failure when expose to temperature which are higher than 1000°C, Due to the thermal expansion mismatch at high temperature.

This problem can be resolved by developing many coatings by mixing various chemical elements with Ytria Stabilised Zirconia. Here we can develop a coating which the mixture of Ytria Stabilised Zirconia and Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) in different chemical composition and deposited on Hastalloy X by plasma spraying technique. The coated specimens are subjected to thermal cycle

and thermal shock analysis and XRD, SEM analysis and using the data we can consolidate the results.

4.3 METHODOLOGY OF COATING

Hastalloy X is selected as the base material, as it can withstand better at higher temperatures. Then the pre-processing process like sample preparation, surface cleaning, surface activation and work is performed.

After the pre-processing method, coating process is performed. This process is performed using Atmospheric Plasma Spray process. Firstly, the bond coat is coated over the substrate material. Then the coating composition process is performed. Here above the bond coat the over coat is coated by the Atmospheric Plasma Spray process. Three types of coatings are followed and the samples are subjected to thermal cyclic and thermal shock test, Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) results are collected of the samples of before and after the test. Using the results the conclusion are analyzed. The detailed explanation is discussed in the upcoming chapters. The full process of the methodology is presented in the below flow chart.

4.4 MATERIALS USED

Hastalloy X which is an Nickel based super alloy is used as the base metal on which the coating is deposited.

The following elements are used as coating materials

- a) Yttria Stabilized Zirconia
- b) Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$)
- c) NiCrAlY powder as bond coat material.

The investigation is illustrated using the flowchart and the detailed experimental procedures in each stage of experiments are explained.

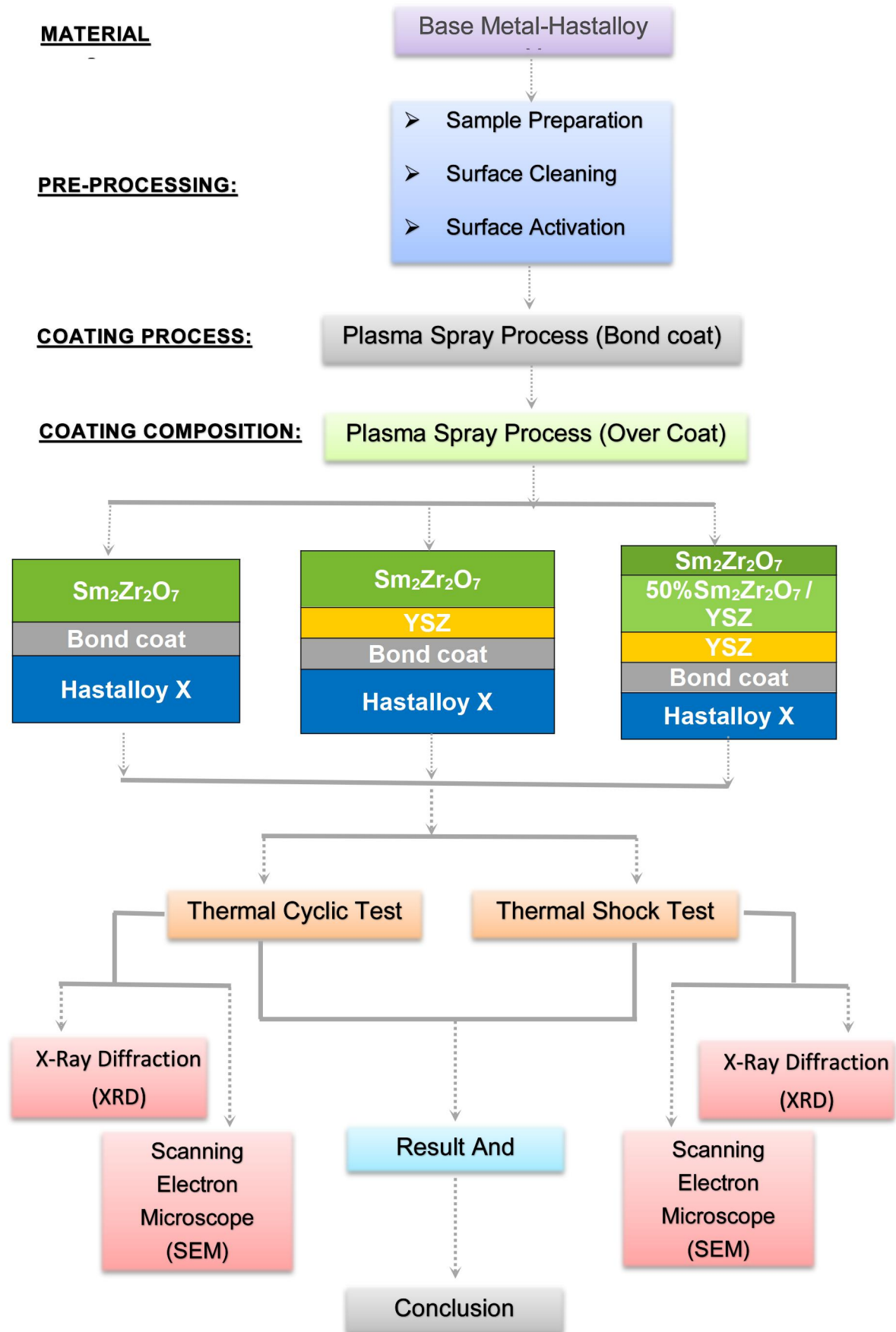


Fig 4.1: Flow Chart For Methodology Of Coating

4.5 PRE-PROCESSING OF COATING

- Hastalloy X which is an NI based superalloy is used as a base metal with an thickness of 5mm.
- The Yttria Stabilized Zirconia, Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$), NiCrAlY as bond coat are brought from **Aplka chemical, Mumbai**.
- The base metal Hastalloy X is cut into 6 pieces which includes circular pieces with diameter 20mm of 27, from **Chozha Tech Engineering** and the dimensions are based on the holding dimension capacity of plasma spraying machine.
- The Plasma spray coating technique is preferred for the deposition of coating substrate which is done at Spray met coating industries, **Bengaluru**.
- The specimens are bond coated with NiCrAlY powder with the thickness of 50 micrometers.
- At high temperature plasma flame the bond coat material in the form of powder is injected, where it is rapidly heated and accelerated into high velocity.
- The hot material impacts on the substrate and rapidly cools forming a coating.
- The bond coating is done to have better adhesion between top coat and substrate.
- After the bond coat, Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) and the mixture of Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) and Yttria Stabilized Zirconia which is in the form of powder are coated into three different sets on the specimens using plasma spray technique.
- The mixture of coated powder on the specimens varies in percentage with a thickness of 300 micrometers.

4.6 COATING TECHNIQUE

Thermal barrier coating is a process in which molten, semi-molten or solid particles are deposited on a substrate. Thermal barrier coatings (TBCs) are a form of protective coating that is applied to high-temperature components such as gas turbine engines and exhaust systems to isolate them from the high temperatures encountered during operation. TBCs can aid in the reduction of thermal stresses while also increasing the overall efficiency and longevity of these components. Consequently, the spraying technique is a way of generating a 'stream' of such particles. Coatings can be generated if the particles that can plastically deform at impact with the substrate, which may only happen if they are molten and sufficiently rapid. TBCs can be applied in a variety of ways, including thermal spraying, electron-beam physical vapour deposition, and plasma spraying. Here we used Atmospheric Plasma Spray as coating technique.

4.7 PLASMA SPRAY COATING

Plasma spray deposition is a single-step method that involves particle melting, quenching, and consolidation. The material to be deposited is injected into a plasma jet, where it is melted and the molten particles contact the substrate surface, generating an adherent coating. The material can be supplied in the form of powder (powder spraying) or liquid (liquid spraying) (wire spraying). The heated substance strikes the surface of the substrate and immediately cools, creating a coating.

The plasma spray technique was performed on three different chemical compositions by varying in percentage as follows.

4.7.1 *Single layer - Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$*

- The Bond coat with 50 micrometers is coated on the base metal Hastalloy X using the plasma spray technique and NiCrAlY is used as the bond coat powder.

- The Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with thickness of 250 micrometers above the bond coat which is the considered as top coat. The coating materials are in the form of powder.

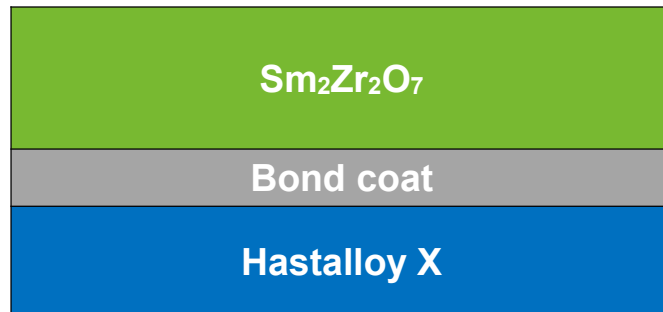


Fig 4.2: Single Layered Coating

4.7.2 Double layer – 7YSZ / Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$

- The Bond coat with 100 micrometers is coated on the base metal Hastalloy X using the plasma spray technique.
- The Yttria Stabilized Zirconia (YSZ) is coated as 2nd layer with thickness of 50 micrometers above the bond coat and the Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with an thickness of 150 micrometers above the YSZ layer.
- This Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) layer is the top coat.
- This type of coating is known as Multi Layered Sandwich form of coating.

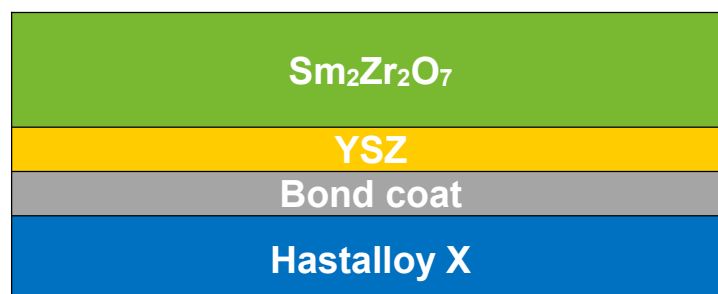


Fig 4.3: Double Layered Coating

4.7.3 Composite Multilayer

- The Bond coat with 50 micrometers is coated on the base metal Hastalloy X using the plasma spray technique.
- Above the bond coat the multi layered sandwich form of coating is done with YSZ is coated with the thickness of 50 micrometers above the bond coat.
- Then above the YSZ coating 50% of YSZ and 50% of Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) mixture is coated with the thickness of 100 micrometers and then Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with the thickness of 100 micrometers above the 2nd layer coating which is the top coat.

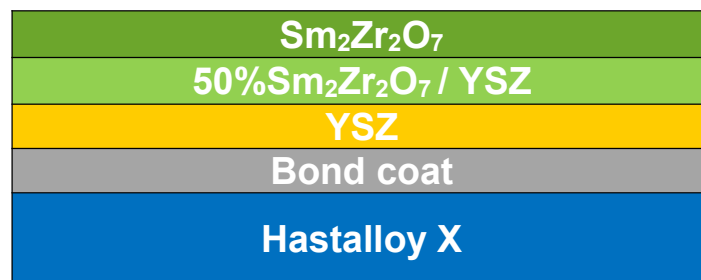


Fig 4.4: Multi Layered Coating

4.8 MATERIAL PREPARATION FOR APS COATING:

Material shaping and sizing in accordance with coating requirements. In the plasma spraying procedure, the gun should require the least amount of powder to accomplish the coating operation (even though the coating surface requires a tiny amount of powder, spraying will occur only if the gun is filled to its minimal required level). Similarly, when it comes to specimen size, the specimen should be able to be held by machine holders during the spraying process. As a result, specimen size should be done appropriately.

The 300micrometers thickness coating is applied to the substrate by utilising the APS technique, which includes bond coat and TBC. In the image below, the

coated illustration is specialised. As stated in the coating methods section, a total of three kinds of coating are applied on the substrate with varying compositions. The difference between the seven types of coatings can be clearly seen in the microscopic test, but the usual visual picture of coated samples can be seen in the figure below.

We have cut the plate into 6 pieces and allowed them to coating operation since we are executing 3 separate sorts of coating operations. Firstly, bond coating is applied in order to adhere the TBC layer for a longer life. TBC should be performed immediately following the completion of Bond coating, as Bond coating loses its ability to adhere after 2 hours of coating. It is also preferable to undertake any cutting operations prior to the coating process because it is difficult to execute cutting operations after TBC. Because the TBC layer we created is non-conductive, the wire cutting axis is unable to pass through it properly.

Nevertheless, due to a technical issue, we attempted to undertake a cutting operation after plasma spraying.

4.9 THERMAL CYCLIC TEST

In order to withstand the high temperature and corrosion resistance the optimum samarium oxide has to be identified. Thermal cycling test was performed on a muffle furnace. Hence three types of composition of YSZ doped with Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) are undergo thermal cycling test which consist of each specimen from each 3 types of total specimens.



**Single Layered
coating**



**Double Layered
coating**



**Multi Layered
coating**

Fig 4.5: Sample images of SZ1, SZ2, SZ3 Respectively

a) Single Layered Coating:

- i. Bond coat of 50 μ m.
- ii. Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) of 250 μ m thickness.

b) Double Layered Coating:

- i. Bond Coat of 50 μ m thickness.
- ii. YSZ of 50 μ m thickness.
- iii. Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) of 200 μ m thickness.

c) Multi Layered Coating:

- i. Bond Coat of 50 μ m thickness.
- ii. YSZ of 50 μ m thickness.
- iii. 50%YSZ + Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) of 100 μ m thickness.
- iv. Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) of 100 μ m thickness.

In order to identify the samarium percentage composition coated sample satisfies the Original Engine Manufacturer (OEM) specification the thermal cyclic test was conducted for each coated specimen. According to the OEM specification, the minimum required number of thermal cycles to pass the thermal cycling test is 217 cycles.



Fig 4.6: Muffle Furnace

The weight loss and the number of cycles till coating starts to peel off were compared for the coated samples. The thermal cyclic test was done in Semco

muffle furnace. The maximum temperature of the muffle furnace is 1300°C. For thermal cyclic process the chosen steady state temperature was 1000°C.

Thermal Shock Resistance (TSR), also known as thermal stress resistance, refers to a fragile material's ability to withstand fast temperature changes without failing. The TSR is determined by the heating circumstances (steady-state flow, transient heating, convection or radiation heating, etc.) and may be represented as the maximum permissible temperature differential in the body. TSR is often assessed for the system of a coating onto a substrate. The majority of the tests imitate coating service circumstances. Coated turbine blades are tested using a conventional thermal cycle furnace test setup.

Thermal Cyclic Fatigue Test was conducted by Furnace cycle test. In Furnace Cycle Test each thermal cycle consist of a 1-hour heat up to the steady-state temperature, and a 10-minute cool down process with in room temperature. The chosen steady state temperature was 1000°C. The peel off determines the starting of the damage.

The specimen kept inside the aluminium boat and kept inside the furnace at 1000°C upto 1hour and cooling down by room temperature upto 10min. This process leads to one complete cycle and according to the OEM specification, the minimum required number of thermal cycles to pass the thermal cycling test is 217 cycles. The weight loss and the number of cycles till coating starts to peel off were compared for the coated samples.

Thermal cyclic fatigue test was conducted on three samples of a particular coated composition. Thermal cyclic fatigue test was carried out confirming to ASTM C 385-58 standards. Initial weights of each coated samples were noted. 3 Coated samples were kept inside the furnace. The coated samples were heated to steady state temperature of 1000°C within 1hour and samples were cooled to room temperature upto 10 minutes. The cooling was done by air. The weight of the coated samples subjected to thermal cyclic test was measured. The cycle was repeated for 250 times. Final weight losses were measured after 217 cycles. The test was performed at Metallurgy Lab, Department of Mechanical Engineering, Sathyabama University.

4.10 THERMAL SHOCK TEST

Thermal shock test consists of rapid change in temperature from extremely high temperature to extremely low temperature and it is also called as accelerated test. The specimen under test is exposed to high temperature for certain time interval and suddenly drops its temperature to extremely very low and these can be used to determine whether the specimen can withstand sudden change in temperature. Thermal shock test is also performed on a muffle furnace. Hence three types of composition of YSZ doped with Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) are undergone thermal shock test which consist of each specimen from each type of total 3 specimens.

The number of shock and weight loss till coating starts to peel off were compared for the coated samples. The thermal shock test was done in Semco muffle furnace. The maximum temperature of the muffle furnace is 1300°C . For thermal cyclic process the chosen steady state temperature was 1000°C .

In the thermal shock test each specimen are kept inside the aluminium boat and kept inside the furnace with high temperature of 1000°C for 10min and cooling temperature at room temperature for 10min interval of time. This process leads to one complete cycle and according to the OEM specification, the minimum required number of thermal shocks to pass the thermal shock test is 713 shocks. The weight loss and the number of cycles till coating starts to peel off were compared for the coated samples.

Thermal shock testing can help identify weaknesses in materials or products that may not be evident under normal operating conditions and by identifying weaknesses in materials or products, thermal shock testing can help reduce product failure rates. It can also identify the change in material properties such as hardness, strength, or ductility and which can lead to the formation of new phases or the development of internal stressors.

The Thermal Shock Resistance is determined by the heating circumstances (steady-state flow, transient heating, convection or radiation heating, etc.) and may be represented as the maximum permissible temperature differential in the body.

TSR is often assessed for the system of a coating onto a substrate. The majority of the tests imitate coating service circumstances.

Initial weights of each coated samples were noted. 3 Coated samples were kept inside the furnace. The coated samples were heated to steady state temperature of 1000°C with 10 minutes and samples were cooled to room temperature upto 10 minutes. The cooling was done by air. The weight of the coated samples subjected to thermal shock test was measured after 713 shocks. The test was performed at IRC, Sathyabama University.

4.11 X-RAY DIFFRACTION

XRD stands for X-ray diffraction, which is a technique used to analyses the crystal structure of materials. XRD works by directing a beam of X-rays onto a sample, which causes the atoms in the sample to scatter the X-rays in different directions. This scattered radiation produces a diffraction pattern that can be analysed to determine the crystal structure of the material. It is a non-destructive technique, which means that it can be used to analyses samples without damaging or altering them. It is also a highly sensitive technique that can detect small changes in the crystal structure of materials.

Both the thermal cycle and shock tested specimens should test for XRD from which the crystal structure can to determined. XRD test is taken at two stages. As coated which mean coated specimen before subjected to testing XRD is taken and after both the test as thermal cycle and thermal shock test done than again XRD is taken. These data exhibits the crystal structure of the specimens before and after the thermal cycle and thermal shock test. During both thermal cycling and shock test the crystallin structure and materials properties and elements of specimen may changes due to the thermal expansion coefficient, phase transition and these can be analysed using the XRD data by comparing with JCPDS file which consist of h,k,l value for many elements with different molecular structures.

All six ascoated specimens before test are subjected to XRD test and data are noted. Then after completing the thermal cycle and thermal shock test, the

specimens are again subjected to XRD test and for which data are noted. Now we compare both the XRD data to analyse the change occur in the specimens.

4.12 SCANNING ELECTRON MICROSCOPE (SEM)

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to create a variety of signals on the surface of solid objects. The signals generated by electron-sample interaction offer information about the sample, such as its outer appearance (texture), substance composition, crystal structure, and polarity of the sample's constituents. Most programmes collect data across a certain section of the sample's face and generate a 2-dimensional image illustrating spatial variations in these qualities.

All 6 specimens should test for SEM which gives us the microscopic image of the coated surface and cross section images can also obtained from SEM which gives the exact images of layers of coating on the base metal. The specimens should be subjected to take SEM in two stages. As coated specimen before thermal cycle and shock test SEM should taken and after both thermal cycle and shock test SEM should taken separately which can help us to analysis image of the all 6 specimens. These images helps us to determine the area of propagated cracks, oxidations which occurs on the coated specimens due to high temperature.

4.13 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDS)

EDS is an abbreviation for Energy Dispersive X-ray Spectroscopy. It is a technique for determining the elemental composition of a material that is used in combination with scanning electron microscopy (SEM). It is an effective method for determining the chemical composition of materials with great spatial resolution and widely employed in subjects like as materials science, geology, among others.

The EDS detector captures X-ray data from each location on the sample while the SEM scans its surface. The energy and intensity of the X-rays are measured, which may be used to identify the sample's elemental makeup and With specialist software, the EDS data may be processed to create an elemental

map of the sample, which depicts the distribution of different elements throughout the sample's surface. EDC test should be taken for all 6 samples to analysis its elemental composition of specimens with respect to their percentage before and after the thermal shock and thermal cycle test. Both the SEM and EDS tests was performed in SRM university lab.

CHAPTER 5

RESULT AND DISCUSSION

5.1 INTRODUCTION:

In this chapter, we are going to proceed with results and discussion of our project.

The data acquired throughout the study is provided in the Results section in a clear manner, utilising tables, graphs and figures to assist show the results. The outcomes are presented in a logical sequence, usually in ascending order of significance.

The interpreted and evaluated results in the context of the research topic and past research in the subject are clearly described in the Discussion section. It explains the relevance of the findings and how they add to the broader knowledge of the study subject.

5.2 SURFACE MORPHOLOGY OF THREE SAMPLES:

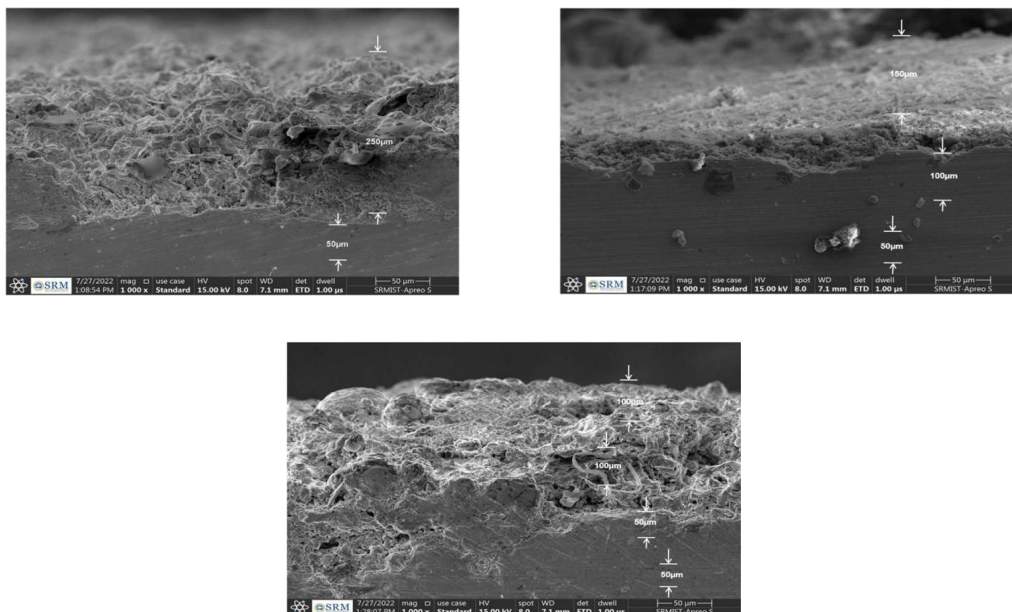


Fig 5.1: Optical images of cross – sectional view of substrate

The illustration depicts a cross-sectional view of the 3 ascoated samples. Figure 5.1 shows the evident creation of the Bond coat and TBC layer. We can observe from the above microscopic picture that TBC is formed by a bond coat on the substrate. The optical picture captures the cross-sectional view seen in the preceding figure.

5.3 THERMAL CYCLIC TEST:

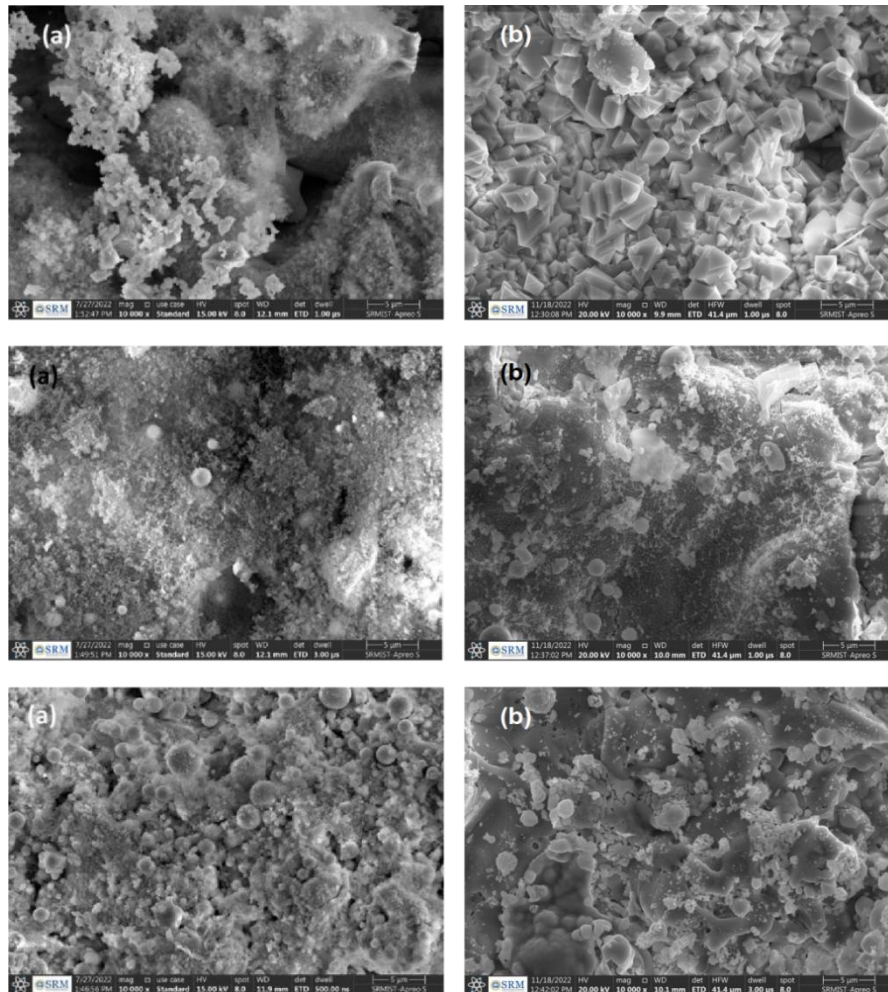


Fig 5.2: (a) Surface Morphology of Ascoated Samples

(b) Surface Morphology of Tested Thermal Cyclic Samples

The figure depicts, the specimens after 217 cycles, subjected to the Thermal cyclic action. The micro structural pictures from the SEM findings can reveal the changes in the specimens. Specimen surface morphology is compared to

as-coated sample surface morphology. From this, we may conclude which coating will perform better when subjected to Thermal Cyclic Test.

5.3.1 Specimen 1: Single Layered Coating

The Bond coat with 50 micrometers is coated on the base metal Hastalloy X using the plasma spray technique and NiCrAlY is used as the bond coat powder. The Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with thickness of 250 micrometers above the bond coat which is considered as top coat. The coating materials are in the form of powder.

- The thermal cyclic test was conducted for the interval of 1 hours with high temperature of 1000°C inside the furnace and cooling at room temperature of 10minutes of time and these defines as one complete cycle.
- The above cyclic process continued for 217 cycles and measures the weight and no changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every shock gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above the 100 cycle the phase transition occurs and as a result small pores are formed.
- The pores get expanded and transformed into small cracks and causes failure of coating after 198 cycles.

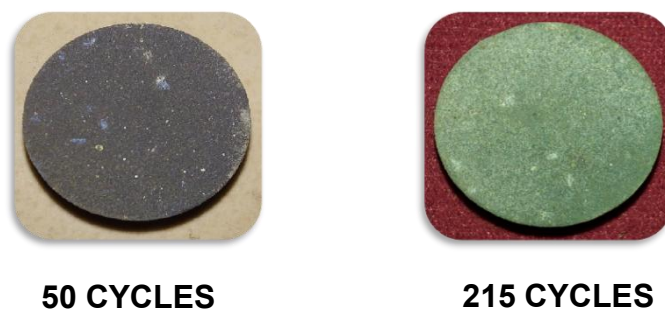


Fig 5.3: SZ1 Sample under gone Thermal Cyclic Test

5.3.1.1 XRD Analysis Single Layered Coating

XRD data of specimen 1 is discussed here. X-ray diffraction pattern for the ascoated TBCs were recorded using the Bruker D8 Advance diffractometer. The pattern was plotted using origin pro software showing the characteristic peaks for the coating.

- The JCPDS database was used to identify the phases present in the coating. The peaks in the XRD patterns were indexed in accordance with the corresponding JCPDS files.
- The peak represents the presence of Sm_2O_3 , ZrO_2 , $\text{Sm}_2\text{Zr}_2\text{O}_7$ which are in Monoclinic, cubic.
- Shows higher amount of monoclinic ZrO_2 , cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$.

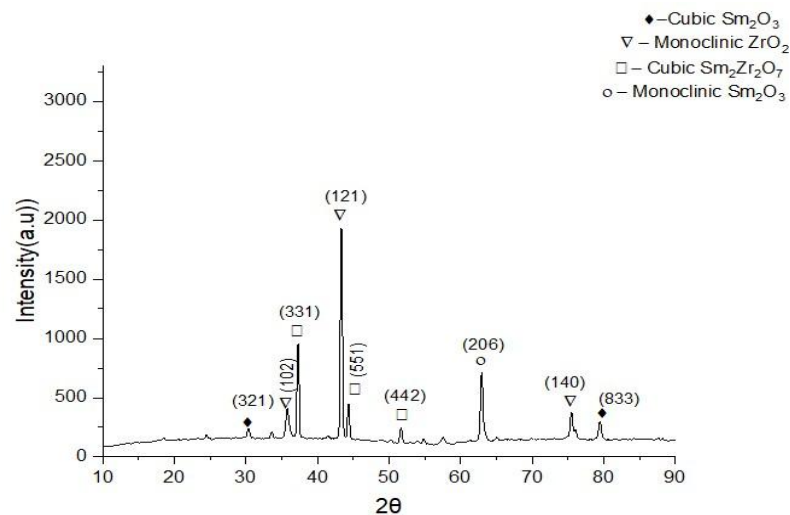


Fig 5.4: XRD Image Of Tested Single Layered Coating

5.3.1.2 SEM Analysis Of Single Layered Coating

SEM data of specimen 1 was discussed here based on fig. The image shows the clear microscopic view of coated power. This image indicates the presence of porosity and some oxide formation on the coating which will act as main cause for failure of coating. Comparing the SEM image of as coated

specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.

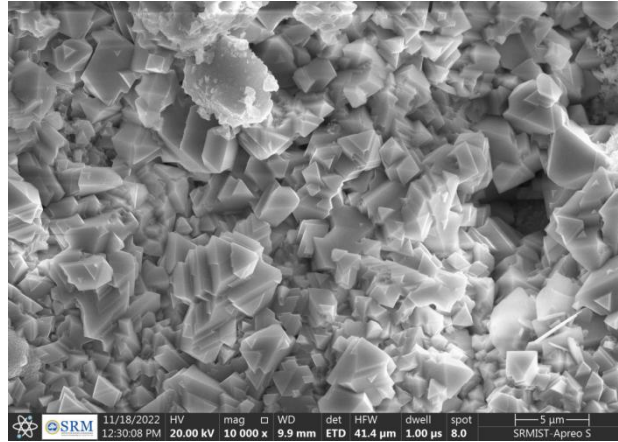


Fig 5.5: SEM Image Of Tested Single Layered Coating

5.3.1.3 EDS Analysis Of Single Layered Coating

- EDS analysis is done using the data in the EDS table and graph shown in fig 5.5.
- The EDS table makes it very obvious what percentage of each element is present.
- Oxygen, samarium, and zirconium may all be clearly identified by examining the SEM and EDS images.
- The table lists the elements current constitutions for the SZ1 sample. We are able to see structural changes brought on by the formation of oxide and severe post-test oxidation of the metal by contrasting the SEM pictures of the As coated sample and thermal cycle SZ1 tested sample.
- The table shows highest percentage of oxygen when compare other elements and lest percentage of zirconium.
- Samarium also exhibits major percentage among the composition of elements after the testing.

Table 5.1: Elements in Tested Single Layered Coating

Element	Weight %
Oxygen	48.76
Zirconium	8.16
Samarium	43.08
Total	100

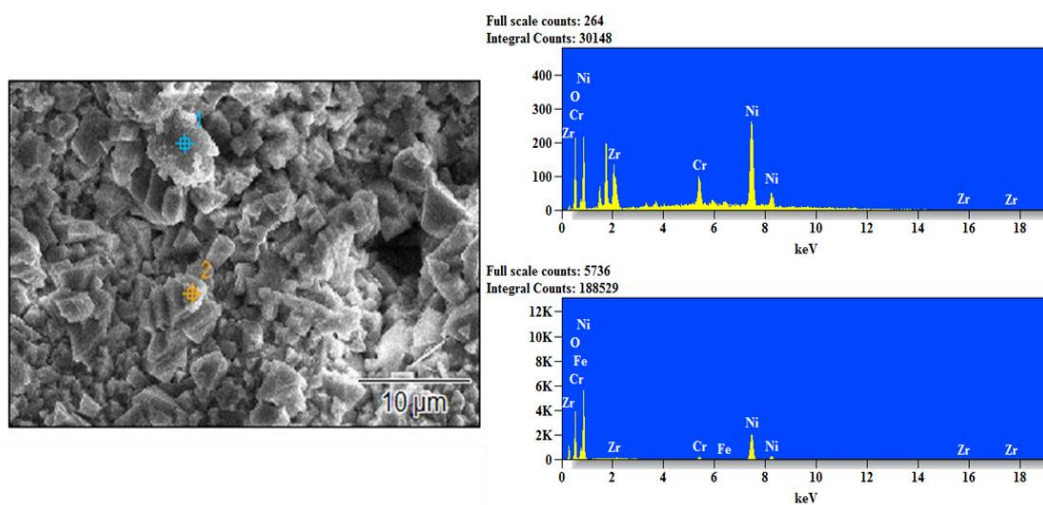


Fig 5.6: EDS spectrum of SZ1 TBC after thermal cycling fatigue test

5.3.2 Specimen 2: Double Layered Coating

The Bond coat with 100micrometers is coated on the base metal Hastalloy X using the plasma spray technique. The Yttria Stabilized Zirconia is coated as 2nd layer with thickness of 50 micrometers above the bond coat and the Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with a thickness of 150 micrometers above the YSZ layer. This Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) layer is the top coat.

- The thermal cyclic test was conducted for the interval of 1 hours with high temperature of 1000°C inside the furnace and cooling at room temperature of 10minutes of time and these defines as one complete cycle.

- The above cyclic process continued for 217 cycles.
- No changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every shock gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above 180 cycles the phase transition occurs and as a result small pores are formed.
- The pores get expanded and transformed into small cracks and causes failure of coating after 210 cycles.



Fig 5.7: SZ2 Sample under gone Thermal Cyclic Test

5.3.2.1 XRD Analysis Of Double Layered coating

XRD data of specimen 2 is discussed here, which gives the crystal structure of materials.

- As per the XRD results, it shows higher amount of monoclinic ZrO_2 , Cubic ZrO_2 , monoclinic Sm_2O_3 , Cubic Sm_2O_3 , Cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$.
- Shows higher amount of monoclinic ZrO_2 , cubic ZrO_2 .
- The intensity of cubic ZrO_2 is higher when compare to other elements intensity.

➤ It shows that higher Cubic, monoclinic crystallin structure is exhibited in specimen 2 after thermal cycle test.

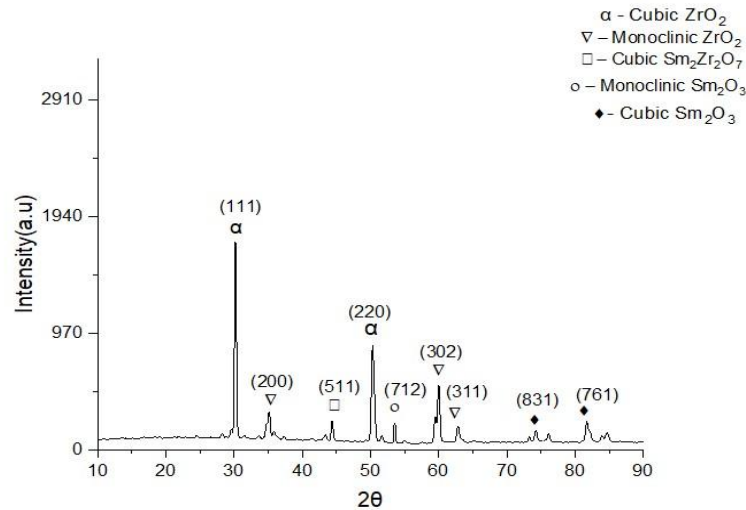


Fig 5.8: XRD Image Of Tested Double Layered Coating

5.3.2.2 SEM Analysis Of Double Layered Coating

SEM data of specimen 2 was discussed here based on fig. The image shows the clear microscopic view of coated power. This image indicates the presence of porosity and some oxide formation on the coating which will act as main cause for failure of coating, but less when compare to first specimen. Comparing the SEM image of as coated specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.

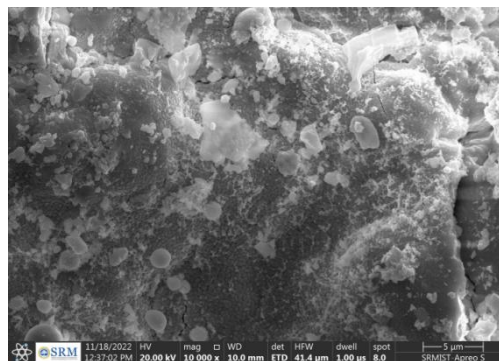


Fig 5.9: SEM Image Of Tested Double Layered Coating

5.3.2.3 EDS Analysis Of Double Layered Coating

- By analyzing the SEM and EDS images we can clearly confirm the presence of Oxygen, Samarium and Zirconium.
- From the EDS table we can clearly know the percentage of each element present.
- For SZ 2 sample the element constitution present is arranged in the below table.
- The table shows highest percentage of oxygen when compare other elements and lest percentage of Samarium.
- By comparing the SEM images of the as coated sample and thermal cycle tested sample, we can visualize some changes in the structure due to the oxide formation but compared to SZ 1 the percentage of oxygen is higher.
- The Samarium exhibits very low percentage and percentage of zirconium slightly higher than specimen 1.

Table 5.2: Elements in Tested Double Layered Coating

Element	Weight %
Oxygen	86.16
Zirconium	13.72
Samarium	0.11
Total	100

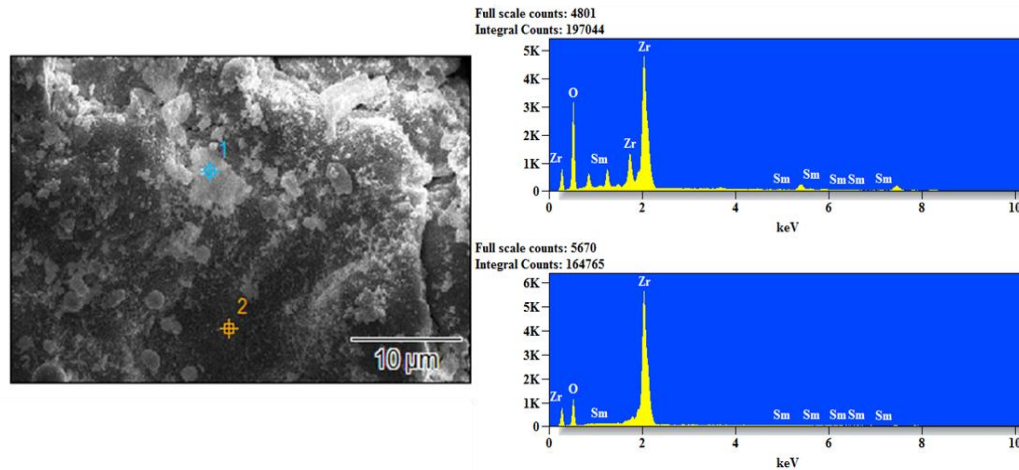


Fig 5.9: EDS spectrum of SZ2 TBC after thermal cycling fatigue test

5.3.3 Specimen 3: Multi Layered Coating

The Bond coat with 50 micrometers is coated on the base metal Hastalloy X using the plasma spray technique. Then above the YSZ coating 50% of YSZ and 50% of Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) mixture is coated with the thickness of 100 micrometers and then Samarium zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$) is coated with the thickness of 100 micrometers above the 2nd layer coating which is the top coat.

- The thermal cyclic test was conducted for the interval of 1 hours with high temperature of 1000°C inside the furnace and cooling at room temperature of 10minutes of time and these defines as one complete cycle.
- The above cyclic process continued for 217 cycles and measures the weight and no changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every cycle gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above the 210 cycle the very small phase transition occurs, but not as compare to specimen 1 and specimen 2.

- This specimen undergoes very less breakage in the topcoat coated layer even after 215 cycles and failure does not occurs.
- So, we say specimen 3 coating can withstand a greater number of cycle when compare to other two coatings.



Fig 5.10: SZ3 Sample under gone Thermal Cyclic Test

5.3.3.1 XRD Analysis Of Multi Layered coating

XRD data of specimen 3 was discussed here, which gives the crystal structure of materials.

- As per the XRD results, it shows higher amount of monoclinic ZrO_2 , monoclinic Sm_2O_3 , Cubic Sm_2O_3 , Tetragonal Y- ZrO_2
- Shows higher amount of cubic Sm_2O_3 .
- The intensity of cubic Sm_2O_3 is higher when compare to other elements intensity.
- It shows that higher Cubic crystallin structure is exhibited in specimen 3 after thermal cycle test.

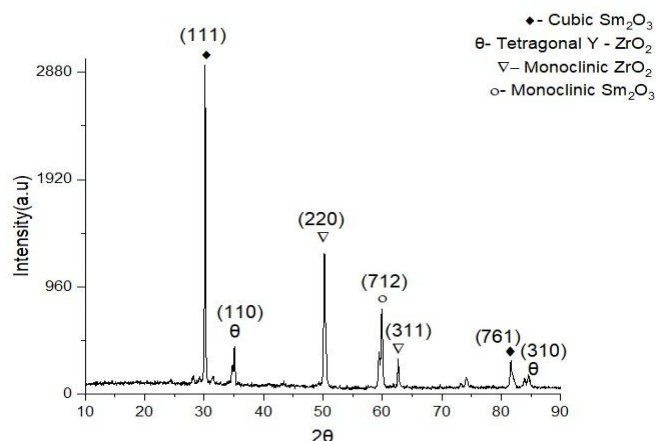


Fig 5.11: XRD Image Of Tested Multi Layered Coating

5.3.3.2 SEM Analysis Of Multi Layered Coating

SEM data of specimen 3 was discussed here based on fig. The image shows the clear microscopic view of coated power. This image indicates the presence of very less oxide formation on the coating but very less when compare to first and second specimen. Comparing the SEM image of as coated specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.

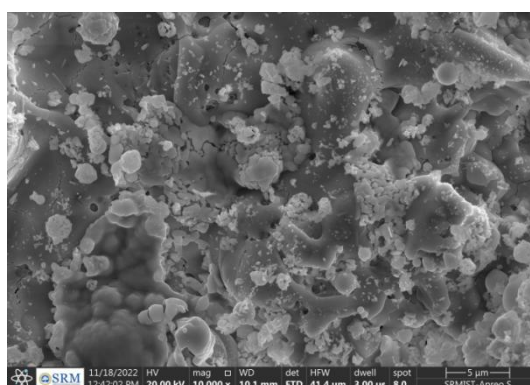


Fig 5.12: SEM Image Of Tested Multi Layered Coating

5.3.3.3 EDS Analysis Of Multi Layered Coating

➤ By analyzing the SEM and EDS images we can clearly confirm the presence of Oxygen, Samarium and Zirconium.

- From the EDS table we can clearly know the percentage of each element present.
- For SZ 3 sample the element constitution present is arranged in the above table.
- The table shows highest percentage of oxygen when compare other elements and least percentage of Zirconium.
- By comparing the SEM images of the as coated sample and thermal cycle tested sample, we can visualize some changes in the structure due to the oxide formation but compared to SZ 1 and SZ 2 samples the structural change is lesser.
- The Zirconium exhibits very low percentage and percentage of Samarium slightly lower than specimen 1.

Table 5.3: Elements in Tested Multi Layered Coating

Element	Weight %
Oxygen	79.99
Zirconium	6.62
Samarium	13.39
Total	100

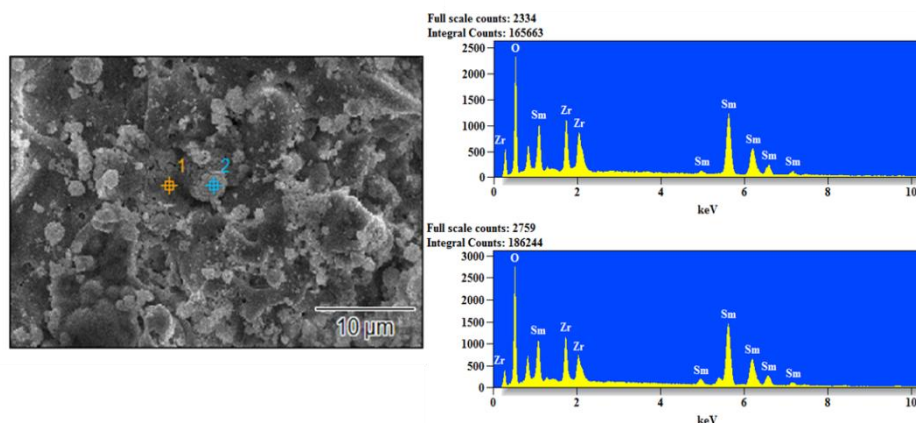


Fig 5.13: EDS spectrum of SZ3 TBC after thermal cycling fatigue test

5.4 THERMAL SHOCK TEST

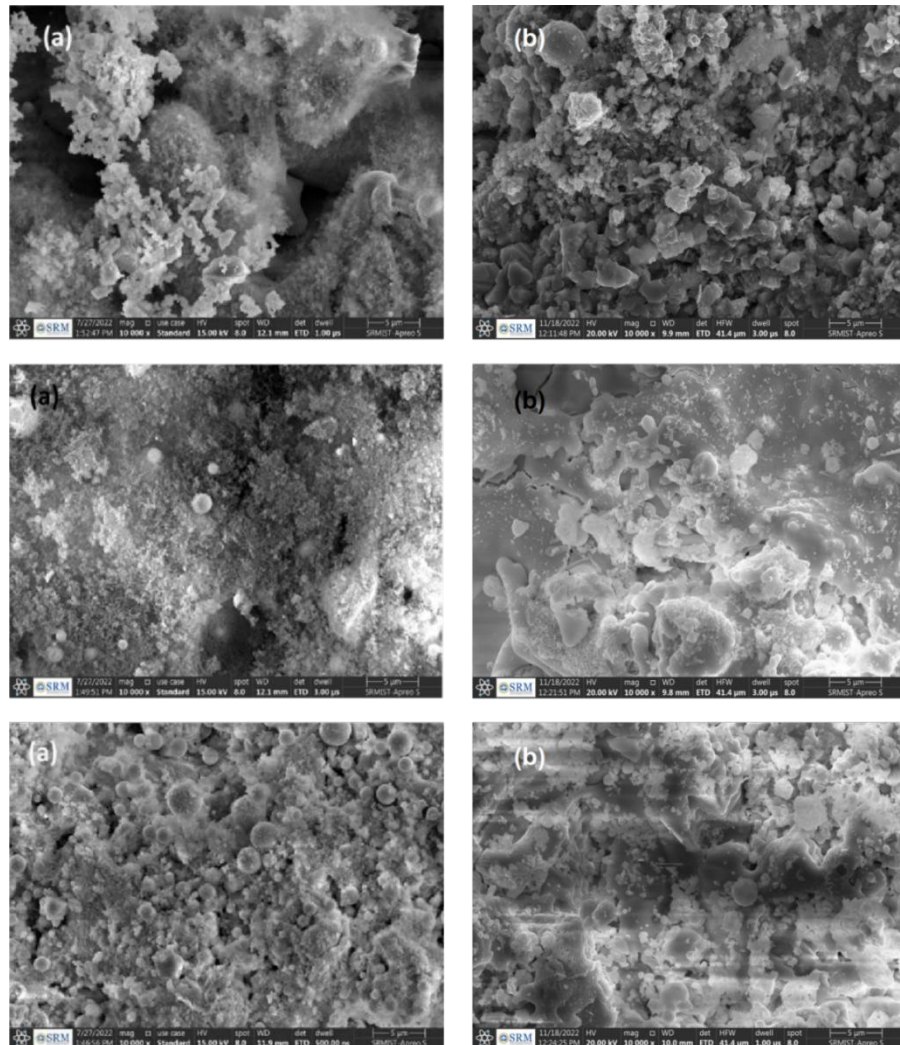


Fig 5.14: (a) Surface Morphology of Ascoated Samples
(b) Surface Morphology of Tested Thermal Shock Samples

The figure depicts, the specimens after 715 shocks, subjected to the Thermal Shock action. The micro structural pictures from the SEM findings can reveal the changes in the specimens. Specimen surface morphology is compared to ascoated sample surface morphology. From this, we may conclude which coating will perform better when subjected to Thermal Shocking Test.

5.4.1 Specimen 1: Single Layered Coating

- The thermal shock test was conducted for the interval of 10 minutes with high temperature of 1000°C inside the furnace and cooling at room temperature of 10 minutes of time and these defines as one complete shock.
- The above shock process continued for 713 shocks and measures the weight and no changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every shock gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above the 570 shock the phase transition occurs and as a result small pores are formed.
- The pores get expanded and transformed into small cracks and causes failure of coating after 615 shocks.



50 SHOCKS



713 SHOCKS

Fig 5.15: SZ1 Sample under gone Thermal Shocking Test

5.4.1.1 XRD Analysis Single Layered Coating

XRD data of specimen 1 was discussed here. X-ray diffraction pattern for the as coated TBCs were recorded using the Bruker D8 Advance diffractometer to analyses the crystallin structure of the specimen 1 after thermal shock test. The pattern was plotted using origin pro software showing the characteristic peaks for the coating.

- The peak represents the presence of Sm_2O_3 , ZrO_2 , $\text{Sm}_2\text{Zr}_2\text{O}_7$ which are in Monoclinic, cubic, tetragonal.
- As per the XRD results, it shows higher amount of cubic Sm_2O_3 , cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$.
- During the Thermal shock test the samarium zirconate coating with the conditions of high temperature has formed Monoclinic ZrO_2 .
- During the Thermal shock test , the samarium zirconate coating pyrochlore $\text{Sm}_2\text{Zr}_2\text{O}_7$ will lose some molecules and break into cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$.

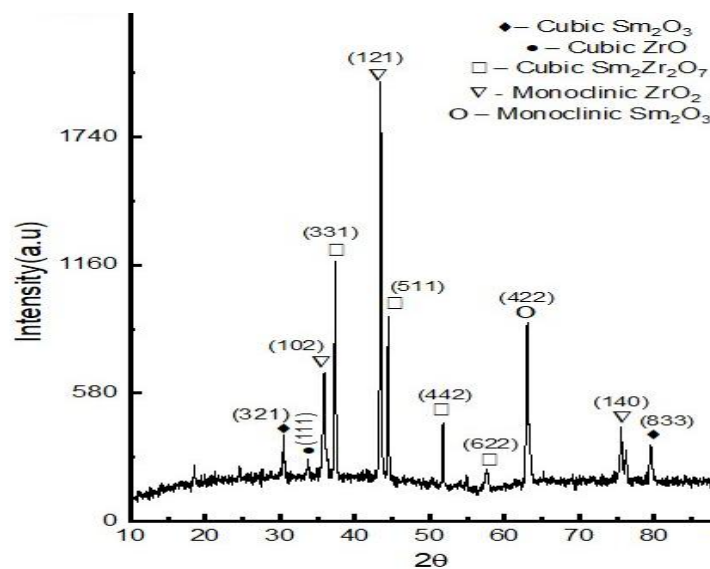


Fig 5.16: XRD Image Of Tested Single Layered Coating

5.4.1.2 SEM Analysis Of Single Layered Coating

SEM data of specimen 1 after thermal shock test was discussed here based on fig.5.14. The image shows the clear microscopic view of coated power. This image indicates the presence of porosity and some oxide formation on the coating which will act as main cause for failure of coating. Comparing the SEM image of as coated specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.

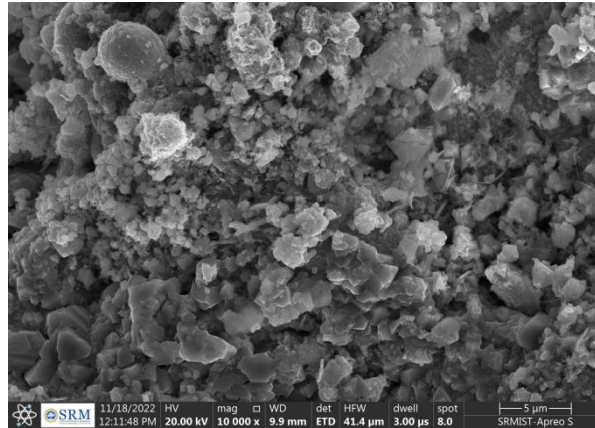


Fig 5.17: SEM Image Of Tested Single Layered Coating

5.4.1.3 EDS Analysis Of Single Layered Coating

- The EDS table makes it very obvious what percentage of each element is present.
- Oxygen, samarium, and zirconium may all be clearly identified by examining the SEM and EDS images.
- The above table lists the elements current constitutions for the SZ1 sample.
- We are able to see structural changes brought on by the formation of oxide and severe post-test oxidation of the metal by contrasting the SEM pictures of the As coated sample and thermal shock SZ1 tested sample.
- The table shows that higher percentage of oxygen is present which indicate the higher formation of oxide on the coated specimen 1.
- The percentage of Zirconium is least when compare to other elements.

Table 5.4: Elements in Tested Single Layered Coating

Element	Weight %
Oxygen	81.56
Zirconium	8.96
Samarium	9.48
Total	100

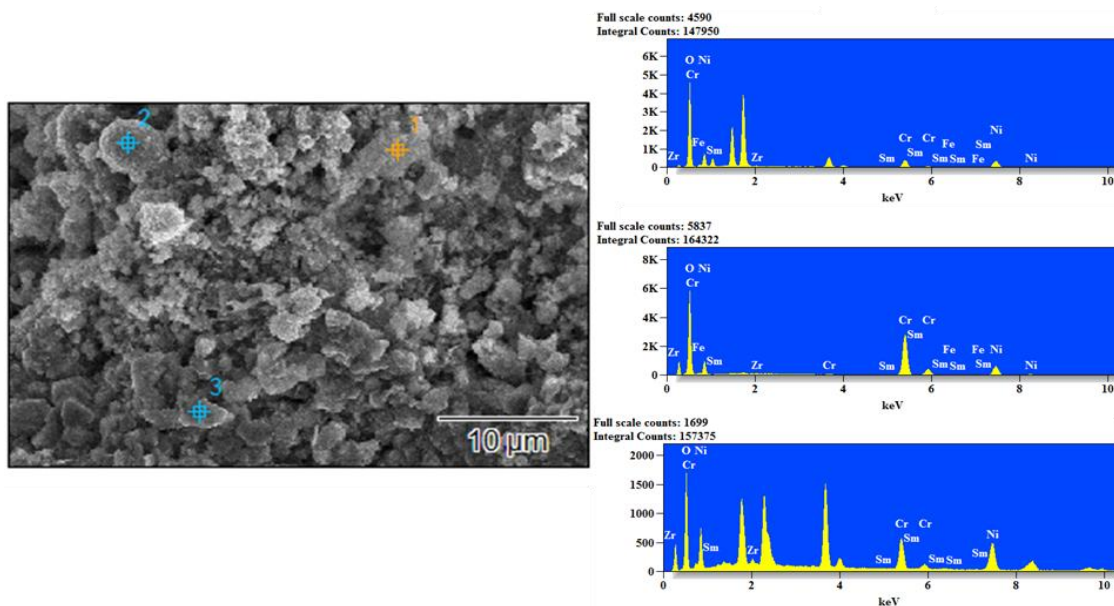


Fig 5.18 EDS spectrum of SZ1 TBC after thermal shocking test

5.4.2 Specimen 2: Double Layered Coating

➤ The thermal shock test was conducted for the interval of 10 minutes with high temperature of 1000°C inside the furnace and cooling at room temperature of 10minutes of time and these defines as one complete shock.

- The above shock process continued for 713 shocks and measures the weight and no changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every shock gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above the 650 shock the phase transition occurs and as a result small pores are formed.
- The pores get expanded and transformed into small cracks and causes failure of coating after 710 shocks.
- When compare to specimen 1 it can withstand higher number of shocks and we can consider this as more suitable for high temperature applications.



50 CYCLES



215 CYCLES

Fig 5.19: SZ2 Sample under gone Thermal Shocking Test

5.4.2.1 XRD Analysis Of Double Layered coating

XRD data of specimen 2 after thermal shock test was discussed here, which gives the crystal structure of materials.

- As per the XRD results, it shows higher amount of monoclinic ZrO_2 , monoclinic Sm_2O_3 .
- During the Thermal shock test the samarium zirconate coating with the conditions of high temperature has formed Monoclinic ZrO_2 .

- The separated Sm_2 molecules from $\text{Sm}_2\text{Zr}_2\text{O}_7$ will react with Oxygen and forms cubic Sm_2O_3 .
- The next present element is the monoclinic Sm_2O_3 is more when compared with other elements.

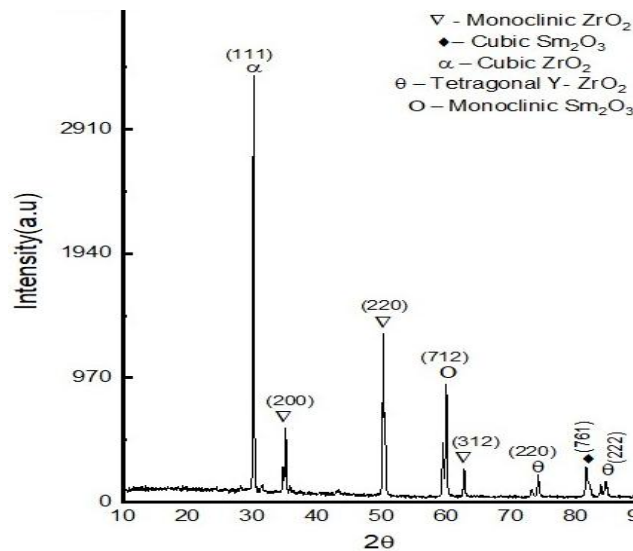


Fig 5.20: XRD Image Of Tested Double Layered Coating

5.4.2.2 SEM Analysis Of Double Layered Coating

- SEM data of specimen 2 after thermal shock test was discussed here based on fig. The image shows the clear microscopic view of coated power.
- This image indicates the presence of porosity and some oxide formation on the coating which will act as main cause for failure of coating.
- Comparing the SEM image of as coated specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.
- The image shows that oxidation formation is less when compare to the specimen 1.

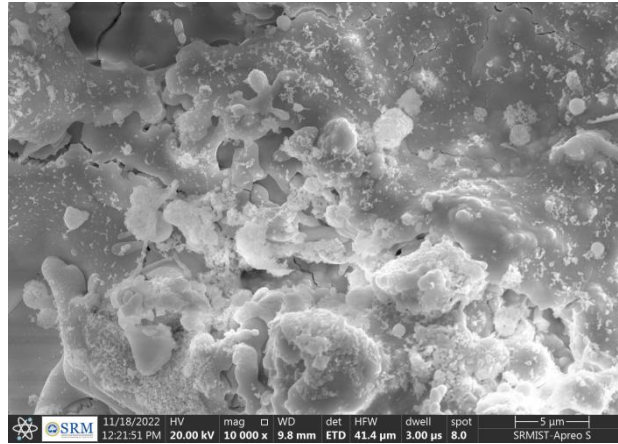


Fig 5.21: SEM Image Of Tested Double Layered Coating

5.4.2.3 EDS Analysis Of Double Layered Coating

- By analyzing the SEM and EDS images we can clearly confirm the presence of Oxygen, Samarium and Zirconium.
- From the EDS table we can clearly know the percentage of each element present.
- For SZ 2 sample the element constitution present is arranged in the above table.
- By comparing the SEM images of the As coated sample and thermal shock tested sample we can visualize some changes in the structure due to the oxide formation but compared to SZ 1 the structural change is lesser.
- The table shows higher percentage of oxygen which indicate the more oxide formation, but lesser than the specimen 1.
- The percentage of Samarium is very low when compare to other elements.

Table 5.5: Elements in Tested Double Layered Coating

Element	Weight %
Oxygen	79.20
Zirconium	12.46
Samarium	8.34
Total	100

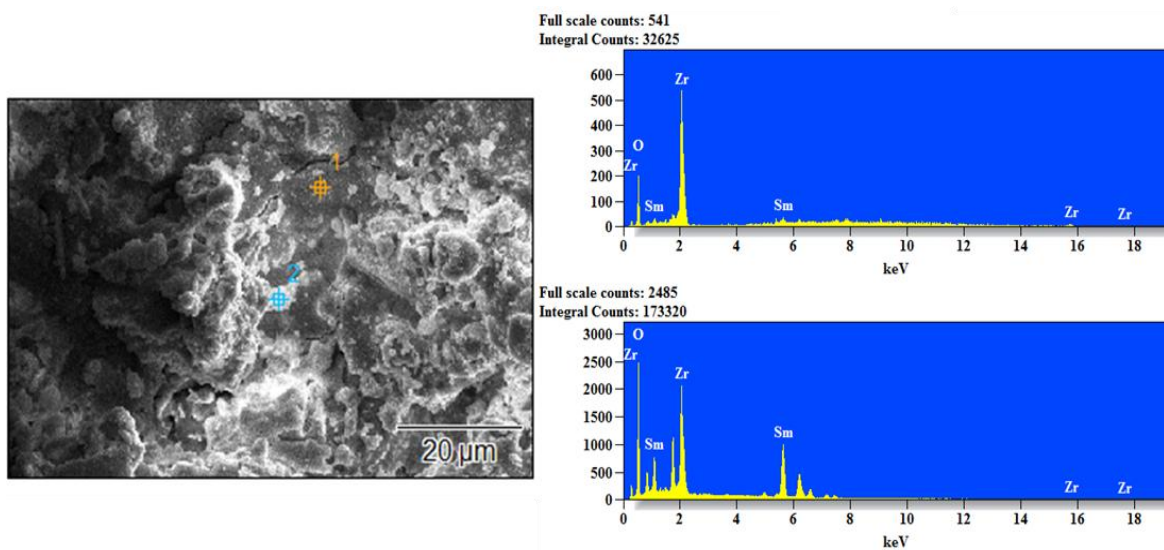


Fig 5.22 EDS spectrum of SZ2 TBC after thermal shocking test

5.4.3 Specimen 3: Multi Layered Coating

➤ The thermal shock test was conducted for the interval of 10 minutes with high temperature of 1000°C inside the furnace and cooling at room temperature of 10 minutes of time and these defines as one complete shock.

- The above shock process continued for 713 shocks and measures the weight and no changes found on the specimen and measured weight also remains same.
- The observation of specimen frequently of every shock gives the detailed Phase transformation and the transitions occurs on the material due to sudden temperature change.
- As above the 700 shock the phase transition occurs which is very less.
- This specimen 3 undergoes very less breakage in topmost coated layer even after 713 thermal shocks.
- When compare to specimen 1 and specimen 2 it can withstand higher number of shocks.
- we can consider this as more suitable for high temperature applications.



Fig 5.23: SZ3 Sample under gone Thermal Shocking Test

5.4.3.1 XRD Analysis Of Multi Layered coating

XRD data of specimen 3 after thermal shock test was discussed here, which gives the crystal structure of materials.

- As per the XRD results, it showed the presence of Tetragonal Y- ZrO_2 , Cubic ZrO_2 , Monoclinic ZrO_2 , Cubic Sm_2O_3 .
- During the Thermal Shock test the samarium zirconate coating with the conditions of high temperature has formed Monoclinic ZrO_2 .

- The separated Sm_2 molecules from $\text{Sm}_2\text{Zr}_2\text{O}_7$ will react with Oxygen and forms cubic Sm_2O_3 .
- Sample (c) shows higher intensity of Tetragonal ZrO_2 when compare to other elements

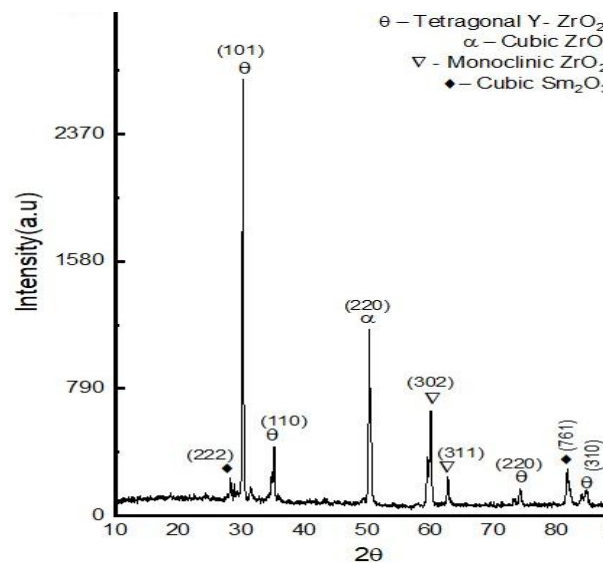


Fig 5.24: XRD Image Of Tested Multi Layered Coating

5.4.3.2 SEM Analysis Of Multi Layered Coating

- SEM data of specimen 3 after thermal shock test was discussed here based on fig.5.20.
- The image shows the clear microscopic view of coated power.
- This image indicates the presence of porosity and some oxide formation on the coating which will act as main cause for failure of coating.
- Comparing the SEM image of as coated specimen before testing and image of specimen after testing gives the details regarding formation of oxide due to high temperature.

- The image shows that oxidation formation is less when compare to the specimen 1 and specimen 2.

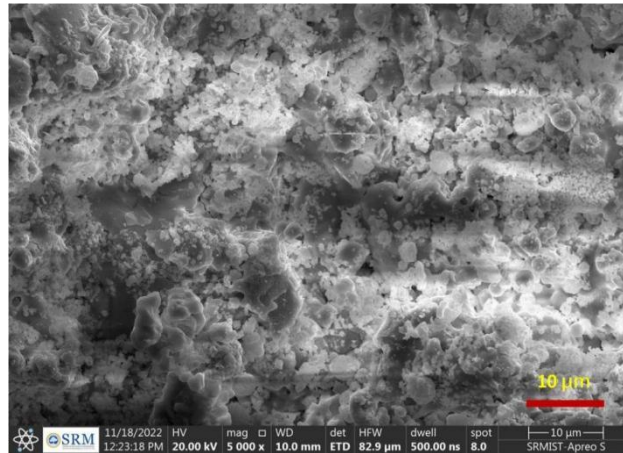


Fig 5.25: SEM Image Of Tested Multi Layered Coating

5.4.3.2 SEM Analysis Of Multi Layered Coating

- By analyzing the SEM and EDS images we can clearly confirm the presence of Oxygen, Samarium and Zirconium.
- From the EDS table we can clearly know the percentage of each element present.
- For SZ 3 sample the element constitution present is arranged in the above table.
- By comparing the SEM images of the As coated sample and thermal shock tested sample we can visualize some changes in the structure due to the oxide formation but compared to SZ 1 and SZ 2 samples the structural change is lesser.
- The table shows that the percentage of oxygen is higher but it is the lowest among three specimens of coating which indicate the less oxidation occurs in specimen 3.
- The percentage of samarium is higher when comparing all three specimens which shows that very less damage take place on the coating.

Table 5.3: Elements in Tested Multi Layered Coating

Element	Weight %
Oxygen	78.46
Zirconium	13.56
Samarium	7.98
Total	100

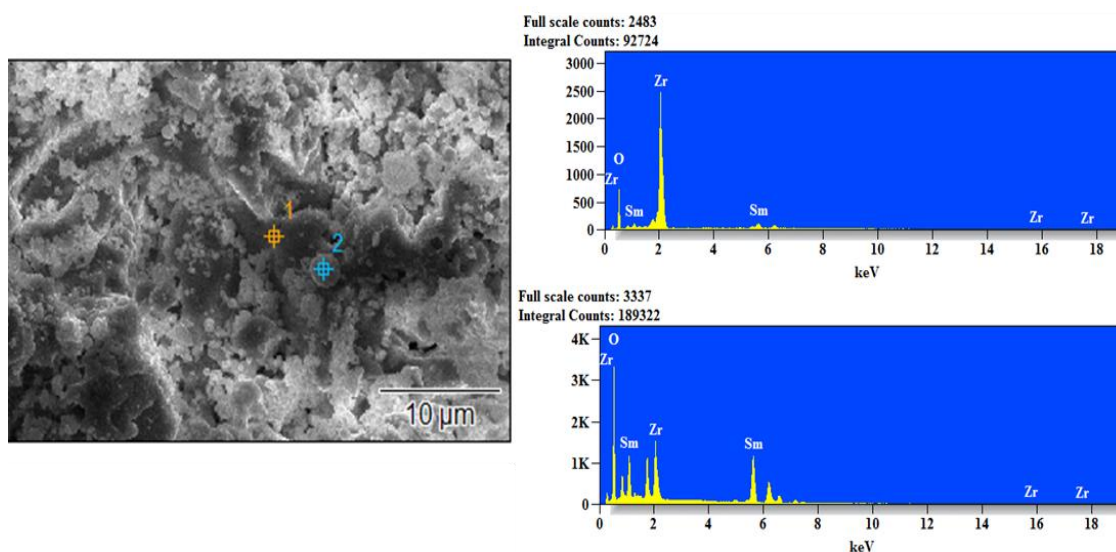


Fig 5.26 EDS spectrum of SZ3 TBC after thermal shocking test

CHAPTER 6

SUMMARY AND CONCLUSION

6.1. SUMMARY

Hastalloy X, which has a wide variety of applications in aircraft turbines, is exposed to thermal barrier coating to strengthen its resistance to high temperatures. At these intense temperatures, i.e., during combustion, the substrate will undergo a reaction with molten salts such as vanadium and sodium sulphate, which develop as bi-products of the fuel utilised in combustion. When Hastalloy X is exposed to those salts at that high temperature, erosion and corrosion will occur. As a result, TBC is the most often used technology for increasing resistivity, and YSZ, which has a resistivity of up to 1200°C, is the most commonly used material for TBC. However, in order to boost temperature resistance over 1200°C, 7YSZ mixed with $\text{Sm}_2\text{Zr}_2\text{O}_7$ is chosen as the TBC material, The current study of investigating the resistance against the thermal cycling and thermal shock fatigue by the three coated specimen.

To test the specimen resistance over the thermal cycling fatigue, the specimen is kept inside the muffle furnace with high temperature of 1000°C for 1hour of time and low temperature of room temperature kept outside furnace for 10minutes of time interval. This change in temperature from high to low is known as one cycle. Due to the transition from extreme high temperature to extreme low temperature the coated specimen could causes damage. The specimens were subjected to more than 210 cycle and among three coated sample one gets withstand up to 217 cycle and other two coating were damaged and peeling of the TBC layer occurred. To test the specimen resistance over the thermal shock fatigue, the specimen is kept inside the muffle furnace with sudden change in temperature from high temperature of 1000°C for 10minutes of time to low temperature of room temperature kept outside furnace for 10minutes of time interval. The failure of TBC layer occurs due to this sudden change in temperature. The specimens were subjected to more than 715 shocks and among three coated

sample one gets withstand up to 715 shocks and other two coating were damaged and peeling of the TBC layer occurred.

6.2. CONCLUSION

In this work, three types of coatings i.e. single layer coating, double layer coating and composite multilayer coating are used as the TBC material and performed by APS technique. The coated specimens were subjected to thermal cycling and thermal shock test inside the furnace. Among the three coated specimens, the third multi-layer coated specimen with stand a greater number of cycles such as 217 cycles and a greater number of shocks such as 715 shocks. Other two specimen (single layer, double layer) were exhibiting the failure and peeling of the coatings. This results that third multi layer specimen is more suitable for high temperature application than single-layer and double-layer coating.

Thermal cycle:

- The subjected single layer coating specimen results in crack formation and peeling of the TBC layer at 198th cycle due to high temperature and low temperature change and no damage occurred to the substrate material. These can compare using the before and after test image.
- The double layer coating specimen results in crack formation and peeling of the TBC layer at 210th cycle due to high temperature and low temperature change and no damage occurred to the substrate material. The SEM image indicated the oxide formed on the specimen and XRD shows the presence of higher monoclinic and cubic Sm_2O_3 and ZrO_2 .
- The multi-layer coating specimen results in no crack formation and peeling of the TBC layer even after 217 cycles and no damage occurred to the substrate material. This results that third multi-layer specimen is more suitable for high temperature application than single-layer and double-layer coating. The SEM image also indicates the less oxide present on the specimen and EDS table shows higher samarium percentage than other two coated specimen.

Thermal shock:

- The subjected single layer coating specimen results in crack formation and peeling of the TBC layer at 615 shock due to sudden change in temperature and no damage occurred to the substrate material. These can compare using the before and after test image. The SEM image and EDS table indicate the higher percentage of oxygen present in the specimen after thermal shock.
- The double layer coating specimen results in crack formation and peeling of the TBC layer at 710 shocks due to sudden change in temperature and no damage occurred to the substrate material. The SEM image indicated the oxide formed on the specimen and XRD shows the presence of higher monoclinic and cubic Sm_2O_3 and ZrO_2 .
- The multi-layer coating specimen results in no crack formation and peeling of the TBC layer even after 715 shocks and no damage occurred to the substrate material. This results that third multi-layer specimen is more suitable for high temperature application than single-layer and double-layer coating.

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