

**Investigation Of Hot Corrosion Behaviour Of
Plasma Sprayed Thermal Barrier Samarium
Zirconate Coatings**

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Engineering degree in Aeronautical

By

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SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with grade "A" by NAAC

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **ABILASH. S** (Reg. No. 39260001), **SRIRAM. R** (Reg. No. 39260039), who carried out the Project entitled "INVESTIGATION OF HOT CORROSION BEHAVIOUR OF PLASMA SPRAYED THERMAL BARRIER SAMARIUM ZIRCONATE COATINGS" under our supervision from June 2022 to April 2023.

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ABSTRACT

In thermal barrier coatings, composite top coat substances that have excellent temperature stability and relatively low thermal conductivity are greatly desired. The two layered structure of Thermal Barrier Coatings (TBC) consists of a bond coat as protective layer and Yttria-Stabilized Zirconia (YSZ) as top layer to provide thermal insulation and to resist oxidation behaviour. Hastelloy X has good resistance to oxidation and corrosion as base metal on that a bond coating of NiCrAlY was sprayed by plasma spray process. Since their introduction in gas turbine engine components, atmospheric plasma sprayed thermal barrier coatings have attracted more curiosity in researchers. Despite the fact even though various new materials are being developed, YSZ remains the industry standard.

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LIST OF SYMBOLS AND ABBREVIATION

ABBREVIATION	DESCRIPTION
TET	Turbine Entry Temperature
TBC	Thermal Barrier Coating
TIT	Turbine Inlet Temperature
YSZ	Yttria Stabilized Zirconia
FCC	Face centered cubic
TEC	Thermal Expansion Coefficient
SOFC	Solid Oxy Fuel Cells
APS	Atmospheric Plasma Spray
EB – PVD	Electron Beam – Physical Vapor Deposition
SPS	Suspension Plasma Spray
DCL	Double Ceramic Layer
HVOF	High Velocity Oxygen Fuel
XRD	X – Ray Diffraction
SEM	Scanning Electron Microscope
EDS	Energy Dispersive Spectrum
CVD	Chemical Vapor Deposition
TGO	Thermally Grown Oxide

SYMBOLS	DESCRIPTION
K	Thermal conductivity
A	Thermal Expansion coefficients
μm	Micron Meter
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

CHAPTER – 1

INTRODUCTION

1.1. INTRODUCTION

When compared to other types of coatings, Thermal Barrier Coatings (TBCs) contain more complicated structures and operate in the extreme high temperature conditions seen commercial turbine engines. A range of structural engineering materials are shielded by coatings against corrosion, wear, and thermal insulation. A low-thermal-conductivity ceramic made of TBCs is used to protect metallic products from the hot gas stream in current gas turbine engines used for aircraft and marine propulsion. The engines with thermal barrier coatings may run at higher gas temperatures when component heating is not as intense. Greater fuel control and longer component service life are the benefits of this.

Hastelloy X, a nickel-based super alloy, was chosen because of its great strength at high temperatures. It has outstanding tensile strength and oxidation resistance up to 2200°F. To improve their resistance against corrosion behaviour of Hastelloy X nickel based super alloy, Thermal Barrier spray coating is used which is operated at high temperatures with various proportions of different chemical compositions on the super alloy. The chemicals used in this process are Samarium Zirconate and yttria stabilized zirconia (YSZ).

In recent years, nickel based alloys are used for aerospace materials because of their low density, withstanding behaviour to high temperatures and high melting point. In order to develop TBC systems or future turbine (gas) engine applications, it is necessary to identify coating materials that can function for extended periods of time and at higher surface temperatures. It is also necessary to comprehend how the performance and lifespan of the coating are influenced by the robustness of different bond-coats and how they interact with the underlying super alloy.

1.2. GAS TURBINE

A gas turbine is a kind of turbine which generates electricity or gives kinetic energy to an aircraft by spinning it with using pressurized gas. 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.

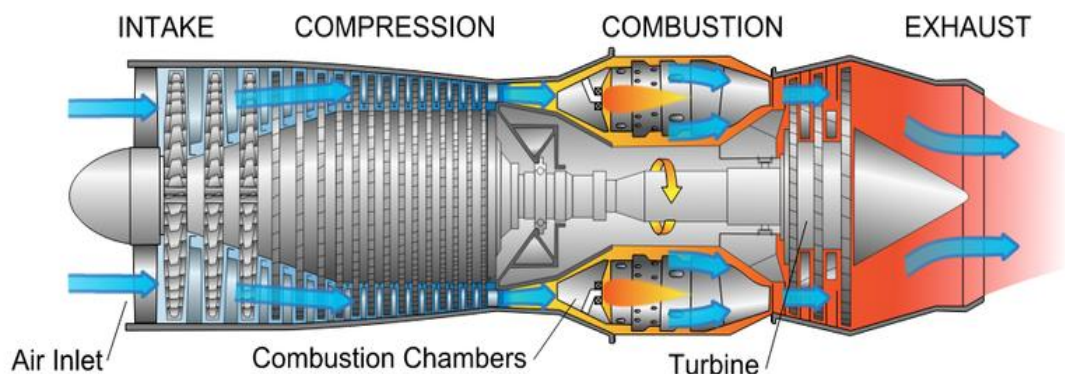


Fig 1.1: Representation of gas turbine engine

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.

1.2.1. SUPER ALLOYS

Super alloys are metals that are far more heat resistant than conventional aircraft structural materials and they can keep their rigidity, durability, hardness, and high stiffness even at considerably higher temperatures. Some of their properties are

- Strong tensile strength
- Thermal resistance is excellent.
- Increased resistance to corrosion
- Coefficient of thermal expansion is less

Super alloys are divided into three types: Nickel, cobalt, and iron-based. Nickel-based super alloys are frequently utilized in extreme temperatures where excellent mechanical strength, creep resistance, and resistance to corrosion are required.

Super alloys are primarily employed in the aerospace for rotor blades, as well as in heat exchanger piping, and commercial gas turbines. Some examples for super alloys are

- Hastelloy
- Inconel
- Waspalloy
- Rene alloys
- Incoloy

1.2.2. NICKEL BASED SUPER ALLOYS

Due to their exceptional mechanical and chemical qualities, nickel-based super alloys find extensive use in the chemical, nuclear reactor, aerospace, and marine sectors. One of the most greatest qualities of nickel super alloys is utilised in jet engines is the excellent resistance to creep and stress fracture at extreme temperature. Nickel's primary properties as an alloy basis are that high phase durability of nickel matrix FCC their ability must be improved by following a range of direct and indirect techniques. Moreover, alloying with chromium and aluminium improves the surface stability of nickel.

Table1.1: Percentile composition of Nickel Based Super Alloys

Alloy	Cr	Ni	Co	Mo	W	Nb	Ti	Al	Fe	C	B	Zr
Waspalloy	19.5	57	13.5	4.3	-	-	3	1.4	2	0.07	-	0.09
Hastelloy X	22	47	1.5	9	0.6	0.5	0.15	0.5	18	0.1	0.008	-
INC-718	19	52	-	3	-	5.10	0.5	0.05	18.5	0.08	-	-
U720LI	16	57	15	3	1.2	-	2.5	2.5	-	0.02	0.01	0.03

1.2.3. HASTELLOY X

Hastelloy X is a nickel-based super alloy with higher strength and increased resistance to oxidation up to 2200°F. It also has exceptional shaping and riveting qualities. Because of the convenience and economy with which HASTELLOY X alloy can be manufactured, due to its outstanding tensile, stress, creep, and rupture strength, it is utilized in a wide range of applications. Because of its unique resistance to oxidising, decreasing, and unbiased atmospheres, they are perfect to use in commercial furnace operations.

Ageing process of this alloy will end in a minor rise in both hardness and toughness, with the impact proportional to the number of hours spent at the ageing temperature.

Table1.2: Physical Properties of Hastelloy X

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

Table1.3: Mechanical properties of Hastelloy X

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

Table1.4: Thermal Properties of Hastelloy X

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

1.3. THERMAL BARRIER COATING (TBC)

Thermal Barrier Coating (TBC) enables advanced turbine engines to run at higher temperatures considerably above the super alloy melting point (-1300°C), hence enhancing engine performance as well as efficiency. Alternatively, if operating temperature is slightly cooler, TBC helps minimize metal temperatures, helping engine components to be more durable. 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 complicated interactions like diffusion, oxidizing, phase change, elastic limit, creep deformation, coefficient of thermal expansion, thermal stability, fracture and sintering occur. Improvements in both the metal base and the ceramic topmost layer are considered. Better results and workability, process developments, and identifying and changing failure mechanisms are all examples of advancements. At high temperature region of the gas turbine, nickel based super alloy with high melting point are used as the turbine blades and vanes. The modern generation of TBC systems can withstand surface temperatures that reach up to 1275°C .

There are two layers in a TBC system. The first layer comprises of bond coat dispersed above the nickel based superalloy. The bond coat is a Ni-based alloy

with several additional additives. The second layer comprises of Yttria-Stabilized Zirconia (YSZ) as top most coat dispersed above the bond coat. The important role of the top most coat is it imparts thermal insulation and in the initial layer, where the bond coat prevents the base alloy from corrosion by developing secondary oxide.

TBC material selection is constrained by certain restrictions like increased melting point, It should not undergo phase change between ambient temperature and working temperature, have low thermal conductivity, match thermal expansion with the metal substrate, stick well to the substrate, and have a minimal sintering rate. However all of the requirements cannot be satisfied by one material, only some of them are able to satisfy.

The coefficient of thermal expansion (TEC) and Thermal conductivity are the most essential qualities for TBC materials selection.

1.4. CERAMIC MATERIALS

A substance that is neither metallic nor biological is a ceramic. It might be either crystalline or glassy, or both. Ceramic materials are basically a mixture of clay, earthen elements, powders and water.

Ceramic materials are classified into types determined by their composition:

- Silicate ceramics: Materials with constitution of both oxygen and silicon are called silicates. The two most common forms of silicate ceramics are alumino silicates and magnesium silicates.
- Oxide ceramics: By definition, they are non-metallic and inorganic compounds that include oxygen, carbon, or nitrogen. Alumina, zirconia, silica, aluminium silicate, and other oxide materials are examples of oxide ceramics.
- Non-oxide ceramics: At very high temperatures and severe thermal shock conditions these ceramics helps to overcome extreme wear and corrosion

problems. These ceramics are used in pharmaceuticals, gas industries, rotatable components, pin for projecting welding metal shaping tools.

- Glass ceramics: These type of ceramics are produced by a controlled crystalline procedure. They are basically polycrystalline materials. They have numerous similarities with both glass and ceramic.

1.4.1. COATING MATERIALS USED FOR HIGH TEMPERATURE

1.4.1.1. Yttria – Stabilized Zirconia (YSZ)

At room temperature, yttria-stabilized zirconia (YSZ) is a ceramic in which the incorporation of yttrium oxide helps to stabilize the cubic crystal structure of zirconium dioxide. Common names for these oxides include "yttria"(ZrO₂) and "zirconia" (Y₂O₃).

Zirconia is a crystalline oxide that occurs in three various crystal forms depending on the temperature. They are: 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 normally forms at exceedingly higher temperatures (>2690 °C), it is stabilized by replacing some of the yttrium ions with zirconium ions across a wide temperature range. This makes the material a conductor of O²⁻ 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.

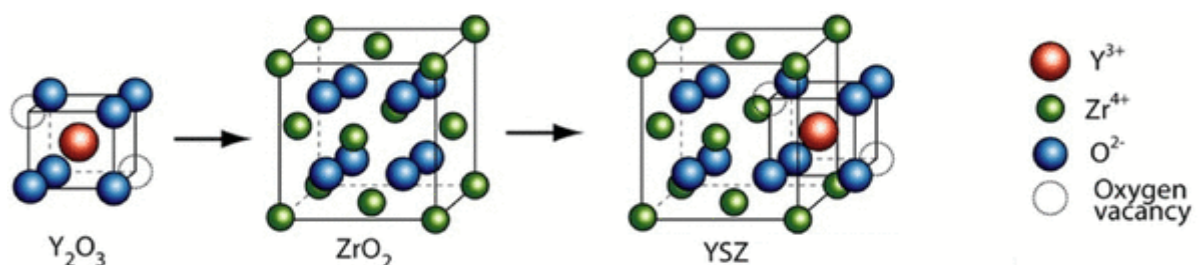


Fig1.2: Formation of YSZ

1.4.1.2. *Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$)*

Samarium Zirconate has good thermal shock protection and high temperature stability. It is a kind of rare-earth zirconate that is frequently utilised in gas turbine engines as a heat shield coating substance. Due to its high melting point, samarium zirconate can be used in high-temperature uses. Additionally, it has a limited thermal transmission, which makes it a superior substance for thermal barrier coatings. The covering serves as a thermal insulator, shielding the engine's metal parts from the high temps that are produced while it is running. Samarium Zirconate is also extremely immune to thermal shock, which is caused by a material's fast heating and cooling and can cause it to fracture or fail. This characteristic is critical in gas turbine engines, which experience frequent temperature shifts while operating.

Samarium Zirconate, in addition to being used as a thermal barrier covering material, has shown promise in other uses such as fuel cells made of solid oxide and electrolytes for high-temperature batteries. Samarium Zirconate can be used as an electrolyte substance in fuel cells made of solid oxide due to its strong oxygen-ion conductivity, which is required for the cell's effective functioning. Overall, because of its exceptional thermal stability, resilience to thermal shock, and distinctive electrical properties, Samarium Zirconate is a potential material for use in high-temperature applications. Its application in gas turbine engines and other industries is anticipated to expand in the future as the demand for high-temperature materials rises.

1.4.1.3. *Lanthanum zirconate ($\text{La}_2\text{Zr}_2\text{O}_7$)*

Lanthanum zirconate is good ceramic material used for TBC because as compared with other thermal barrier coating methods, it has reduced thermal conductivity and greater thermal stability. Comparing with YSZ, LZ has many advantages for TBC operations like, the phase transformation from ambient temperature to its operating temperature does not occur, it has high sintering resistance, it shows a lower thermal conductivity and the key benefit is that LZ has

reduced ionic dispersibility, which prevents corrosion of the bond coat and substrate. The major disadvantage of LZ is it has lower CTE value compared to bond coat and substrate CTE values which do not match. The methods for fabricating LZ powders are co-precipitation method, solid state reaction method and sol-gel method. The LZ compounds are stable at room temperatures, but a change occurs at higher temperatures, where the compounds undergo a structural transition from pyrochlore to defective fluorite. The LZ is deposited over the substrate by Atmospheric Plasma Spraying (APS) process. The APS coated specimens have lesser thermal conductivity when compared with EB-PVD coated specimens. In one research they 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 increased fracture toughness, increased hardness, decreased Young's modulus of rigidity. By the result of thermal cycling behaviour Double ceramic layer based LZ thermal barrier coatings possess higher residual stress even though the fracture resistance is lesser than YSZ.

1.4.1.4. Neodymium Magnesium Aluminium Oxide ($\text{NdMgAl}_{11}\text{O}_{19}$)

A single phase with magnetoplumbite structure was seen in $\text{NdMgAl}_{11}\text{O}_{19}$ ceramic synthesised by technique of solid-state reaction at 1973 K for 10 hours in ambient temperature. YSZ is confined to applications with temperatures less than 1473 K. The phase transition and hardening of YSZ coatings at temperatures over 1473 K result in a decrease in strain tolerance, a rise in Young's modulus, and a volume change of the coating, all of which are detrimental to TBCs. The XRD patterns obtained from the ceramic after 10-50 hours of heat corrosion in air at 1223 K shows three phases are formed as a result of the reactions: Al_2O_3 , NdVO_4 , and MgAl_2O_4 . The XRD analysis revealed no substantial $\text{NdMgAl}_{11}\text{O}_{19}$ phase after the hot corrosion test. By atmospheric plasma spraying, In carbon fibre reinforced silicon carbide ceramic matrix (Cf/SiC) composites, triple-layer ceramic coatings based on the Si bonded layer, Yb_2SiO_5 intermediate layer, and NMA topmost layer

were developed. Thermal stress tests were performed on the coatings at 1573 K, 1673 K, and 1723 K, respectively. The failure mechanism of the Si/Yb₂SiO₅/NMA coating was shown to be closely related to the thermal shock temperature. Under 1673 K, coating failed owing to a mismatch in CTE between the coating and the substrate, an interaction between Yb₂SiO₅ and NMA at the contact, poor sintering characteristics of Yb₂SiO₅ and NMA, and the substrate on its own. Because the temperature was higher than the melting point of the silicon, the coating fractured after only one thermal shock cycle at 1723 K.

1.4.1.5. Neodymium oxide (Nd₂O₃)

Thermal stress tests cause the creation of holes on the coating surface, which grow into cracks and cause coating degradation. These may be reduced by adding Nd₂O₃ particles, and the coatings grew more compact and the size of the small pores on the coatings' surfaces increased while the quantity of small pores decreased. Also, when the quantity of Nd₂O₃ particles increased, the cracks shrank and eventually disappeared, suggesting that the coating's capacity to endure thermal shock could be considerably improved by incorporating Nd₂O₃ particles into the electrolytes. The coating hardness may be enhanced by adding Nd₂O₃, and the coating thickness can be increased and subsequently lowered based on the concentration of Nd₂O₃. The modified Nd₂O₃/doped YSZ coatings shows increased thermal cycling endurance and resilience to thermal shock when compare to others. The major cause of thermal shock failure is removal of inner layer of the ceramic layer. The coating's brightness rose as the rare earth oxide doping level increased, and the spectrum emitted by the Nd₂O₃/modified YSZ coating after the thermal shock test lowers and exhibits the t' phase as the phase structure. When the concentration of Nd₂O₃ increases, the phase change of zirconolite-2M to zirconolite-4M increases, the size of the granule of the crystalline phase reduces, and the production of oxyapatite stage occurs owing to the high doping level of Nd₂O₃, and the chemical durability also increases.

In future MOS technology, Nd₂O₃ with high-K dielectric material is a serious concern that must be continually monitored. The Nd₂O₃ layer created by the low-cost sol-gel dip coating displays robust insulating properties for future

microelectronic applications.

The $\text{SiO}_2/\text{Nd}_2\text{O}_3$ coating was laser ablation resistant and effectively protected the C/C substrates from degradation and thermal deterioration. Thermal and chemical ablation damage were two forms of laser-ablation damage that might be reduced by increasing the reflectivity of the coated surface. Because of its higher ability to reflect light, the $\text{SiO}_2/\text{Nd}_2\text{O}_3$ coating was more effective in protecting against laser damage, significantly extending the service life of the repaired region, and successfully preventing secondary damage.

1.4.1.6. Cerium Zirconate (CeZr_2O_7)

Using a solution-assisted combustion synthesis method, cerium-zirconate powders were created, and they were then calcined at 1123 K for 8 hours to create granules with a rough crystalline structure. Studies of Scanning Electron Microscopy properties were followed in order to evaluate the surface 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 $\text{Ce}_2\text{Zr}_2\text{O}_8$ 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 $\text{Ce}_2\text{Zr}_2\text{O}_8$ 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) because of the higher temperature of the Al_2O_3 layer that formed is the base. The use of coatings on superalloy substrates for a variety of internal and external applications that were influenced by temperature is well known. Highly oxidising conditions demand coatings that are resistant to spalling. Coatings made of ceramic with higher wear and hardness resistant that is subjected to corrosive and heat situations compared to metallic materials, they have a number of advantages due to their low densities. A ceramic surface layer has much lower heat conductivity than a metallic layer. 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). 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. Considering that the majority of the prior studies utilised lowering temperatures of 1573 K or less In this work, the authors demonstrate that 1300°C is insufficient to support the pure phase pyrolysis of CZ and $\text{Ce}_2\text{Zr}_2\text{O}_8$, but at much higher temperatures. To ensure that both phases are pure, a temperature of 1773 K is needed. The primary responsibilities of Materials based on CeO_2 are catalysts to balance the quality of oxygen in exhaust gases by preserving and releasing flowing between the engine's lean and rich operation, respectively. The term "oxygen storage" appropriately describes this oxygen exchange of CeO_2 capacity" (OSC) (OSC).

1.5. COATING TECHNIQUES

The technique of creating a thermal barrier coating (TBC) involves impacting melted or soft particles upon a substrate. Modern TBCs are essential for preventing oxidizing and heat corrosion of engine components 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 an internally cooled gas turbine blade with a relatively low thermal conductivity material. 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 method, Detonation spray method, Cold Spray coating method, Air Plasma spray method, ESVAD, EBPVD, HVOF and SPPS. Among all above coating technologies, Air plasma spraying method is the vastly used technique for the coating purpose.

1.5.1. ATMOSPHERIC PLASMA SPRAY TEST (APS)

For TBCs, Atmospheric Plasma Spray (APS) is a well-liked covering method. The ceramic substance is heated using a plasma torch before being sprayed onto the

base. High porosity coatings from APS are advantageous for heat shielding but may have diminished mechanical qualities. The plasma spray procedure involves spraying melted or heated softened material onto a surface to form a coating. A very hot plasma flame is used to quickly heat and accelerate powder, which is then injected into the flame. Plasma spray is a method where in a single process particle heating, quench, and consolidating are combined. In this method, the material to be deposited is injected into a plasma jet, where it is melted and the melted particles impact the substrate surface, generating an adhesive covering. The plasma spray cannon is made up of a copper anode and a tungsten cathode that are both water cooled. Plasma gas travels round through both the cathode and anode, which is formed like a narrowing nozzle. The arc's resistive heating drives the gas to reach high temperatures, causing it to split and ionise to produce plasma. The main advantages of this method is, it has potentially low complexity setup, also a wider range of coating materials can be used and coating process is fast.

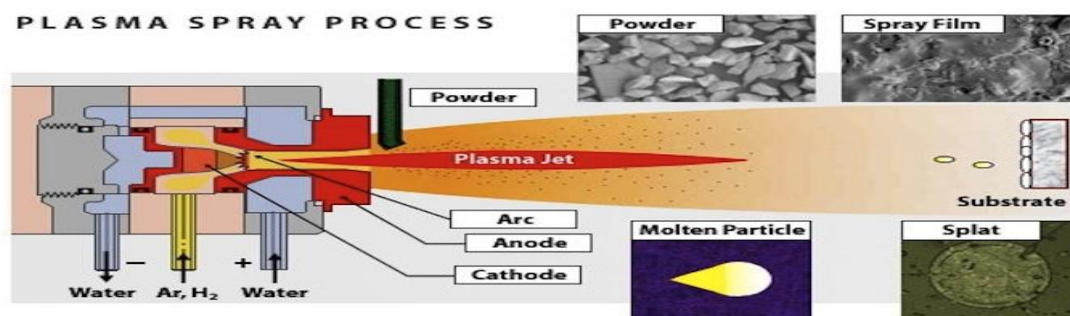


Fig1.3: Representation of Plasma spray process

1.5.2. ELECTRON BEAM PHYSICAL VAPOR DEPOSITION (EB-PVD)

In the EB-PVD process, an electron beam is utilized to increase a porous ingot of the coating material's temperature in a vacuum chamber over the melting point to about 3500°C. For the ingot, porosity is needed so that it can withstand the fast heating by electron beam. Condensation forms the coating while the substrates rotate or move in a planetary motion. Ceramic top coatings for TBCs produced

using the EB-PVD process are composed of loosely linked, columnar granules and have a high degree of mechanical flexibility. The lack of major splat boundaries normal to the heat flow direction indicates that these coatings exhibit relatively high levels of thermal conductivity. Prior to application to the base, the ceramic material is evaporated by an electron stream in the EB-PVD process. EB-PVD creates extremely dense coatings with excellent tensile and thermal properties.

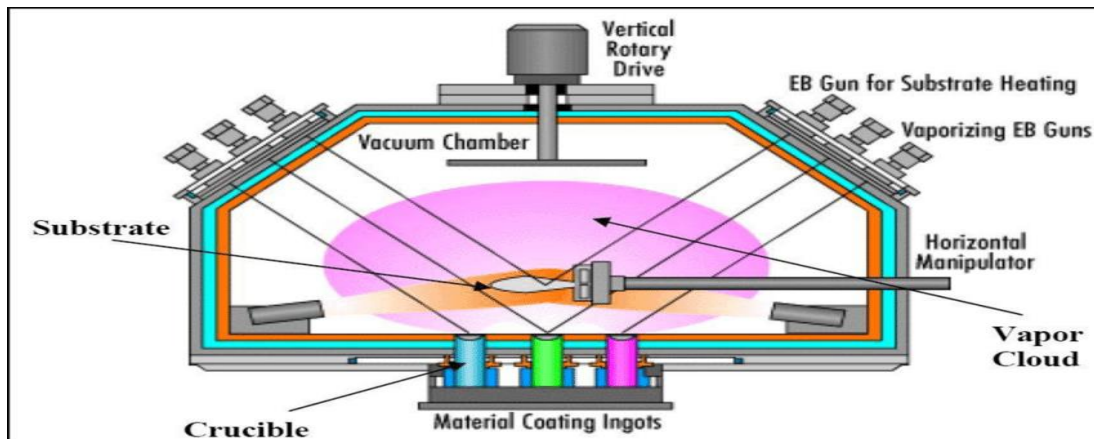


Fig1.4: Representation of EB-PVD process

1.5.3. HIGH VELOCITY OXYGEN FUEL PROCESS (HVOF)

High Velocity Oxygen Fuel (HVOF) coating is a thermal spraying method used to enhance the surface of parts. HVOF spraying functions by combining fluid fuel and oxygen, which is then pumped into and fired in a combustion chamber. The resulting gas has extremely high pressure and temperature and will be expelled at supersonic speed by the nozzle. A stream of hot gas and powder is aimed at the surface to be coated. The thick coating generated has low porosity, higher binding strength, and a variety of benefits, including resistance to corrosion. Using a combustion process, the HVOF method produces a high-velocity stream of gas and powdered ceramic substance. The base is then covered with a thick, high-quality coating after the powder has been de-deposited onto it.

Benefits of HVOF coating:

- Due to greater particle impact velocity it has increased density and decreased porosity.
- It has superior wear resistance due to stronger and tougher coatings.

Drawbacks of HVOF coatings:

- HVOF sprayed coatings are exceedingly complex, with their characteristics and morphology influenced by multiple processing factors.
- Because to the necessity for tight size dispersion, powder sizes are limited to a range of around 5 - 60 μ m.
- This coating method requires very well-experienced and high-quality employees to ensure proper functioning and provide uniform coating quality.

High Velocity Oxy-Fuel Process

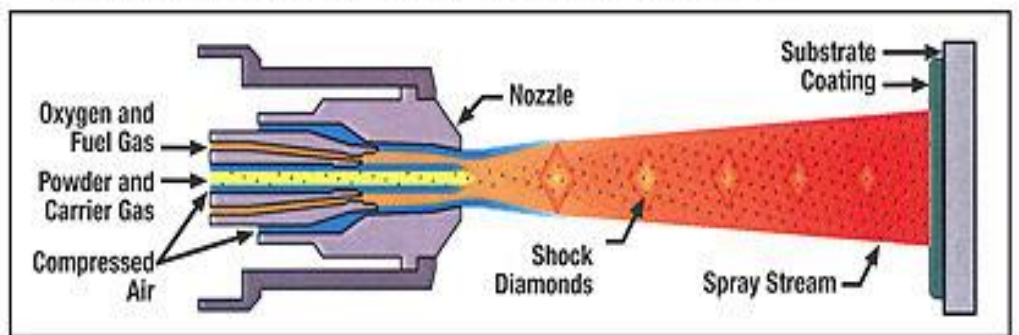


Fig1.5: Representation of HVOF process

1.5.4. ELECTRON BEAM - DIRECT VAPOR DEPOSITION (EB - DVD)

The system makes use of a continually operating axial e-beam gun that has been modified to work in a lower vacuum condition, as well as a He-O₂ feed gas jet with changeable composition. In this process, the vaporised material is compacted in the carrier gas jet and rapidly deposited onto the substrate, resulting in excellent material use efficiency. 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.

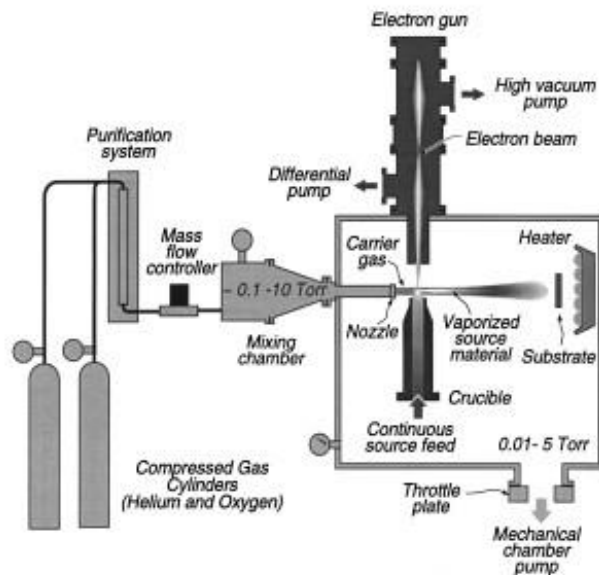


Fig1.6: Schematic representation of EB-DVD process

1.5.5. 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.

1.6. 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.

1.7. 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.

1.8. HOT CORROSION

Hot corrosion test is a type of testing used to assess the susceptibility of materials to corrosion at high temperatures. It is commonly used in the aerospace, power generation, and marine industries, where materials are exposed to elevated temperatures and harsh conditions. When materials are exposed to extreme temperatures, hot corrosion develops, typically above 600 °C, and corrosive environments containing molten salts or other corrosive species, such as sulfur, vanadium, or sodium. The corrosion can cause significant damage to components, leading to reduced performance and increased maintenance costs. In hot corrosion testing the samples of the material to be tested are exposed to elevated temperatures and corrosive conditions for a specified period of time, typically several hundred hours. The corrosion rates that develop on the material's surface are subsequently examined using a variety of analyzing methods. The outcomes of hot corrosion testing can reveal important details about how well materials work in hot, corrosive environments. With the aid of this knowledge, materials for use in particular uses can be better chosen, and coatings or other protective measures can be created to prevent or lessen the effects of hot corrosion. There are several ways to test for hot corrosion, including electrochemical testing, burner apparatus testing, and furnace testing. The type of material being tested, the corrosive species involved, and the intended testing conditions are some examples of variables that influence the method choice. Type-I hot corrosion and Type-II hot corrosion are the two forms of hot corrosion.

1.8.1. *Type-I hot corrosion*

Type I hot corrosion occurs in the 800°C to 900°C temperature range, with the melting temperature of the salt deposit acting as the minimum level. The presence of salt impurities causes molten deposits to form, which degrade the protective coating found on the surface of some metals. When the blade surface is coated with liquid sodium sulphate, type I hot corrosion occurs; solid or gaseous sodium sulphate does not produce corrosion. Many factors determine the temperature range at which sodium sulphate liquefies. Type 1hot corrosion can be reduced by using protective coatings, material selection, and optimizing working conditions. High-temperature alloys and coatings that can withstand the corrosive assault of

salt deposits can assist Increase the lifespan of components that are subjected to high temperature combustion gases.

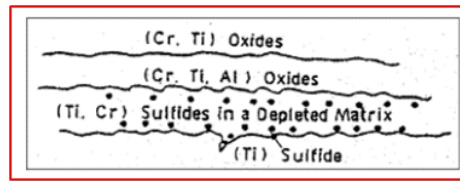


Fig1.7: Type-I hot corrosion

1.8.2. Type-II hot corrosion

Type II hot corrosion can be reduced by using materials and coatings that resist sulfate-rich deposits. High-temperature alloys and coatings that can withstand the corrosive assault of sulfate-rich deposits can assist to improve the durability of components that are subjected to high temperature combustion gases. Optimizing operating parameters such as temperature, pressure, and the composition of the combustion gases, in addition to material selection and protective coatings, can assist to prevent Type II hot corrosion. Appropriate maintenance procedures, such as frequent cleaning and examination of the components, can also aid in the early detection and prevention of corrosion.

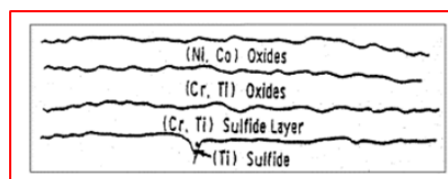


Fig1.8: Type-II hot corrosion

CHAPTER – 2

LITERATURE SURVEY

2.1. INTRODUCTION

The present study is an attempt and research on the best TBC material for providing increased resistance to heat corrosion that happens during combustion in aviation applications. Due to increasing temperatures, particularly higher Turbine Inlet Temperatures at turbines in aviation engines. The interaction with molten salts produced by the burning of the fuel causes erosion and oxidation of the turbine blades and vanes. Salts such as sodium sulphate and vanadium oxide have a greater impact on turbine blades and vanes. Yet, numerous researchers have contributed to creating the most effective materials and technologies to satisfy that challenge. We endeavoured and explored the ideal TBC composition to tackle the problem of hot corrosion-induced erosion and degradation.

2.2. LITERATURE OF THERMAL BARRIER COATING

Mehboob et al (2020) conducted an investigation on Thermal barrier coating degradation mechanisms and techniques for prolonging their lifespan. TBCs with many materials and layers are used to keep turbine engines safe from hot gases. Several variables impact the lifespan of YSZ-TBCs when operated at increased temperatures, includes residual stress, high-temperature sintering, the effect of CMAS, phase change, and TGO formation due to bond coat oxidation. All of these factors combine to reduce the lifespan of YSZ-TBCs at extreme temperatures. Moreover, YSZ-TBCs fail to offer long-term protection for high-temperature turbine engines.

Liu Q et al (2019) investigated about the ceramic composites aero-engine thermal barrier coatings of yttria stabilised zirconia. They mentioned that several desired features must be present in ceramic materials for TBCs, such as increased melting point, lower thermal conductivity, in the operating temperature range, matching CTE, strong adhesion behaviour, low sintering rate. TBCs' most critical features are CTE and thermal conductivity.

B. Liu *et al* (2019) in this paper summarises the commonly utilised methodologies and new advances in researching for TBC materials that are successful in both theoretical and testing approaches. Theoretically, density functional theory has been successfully applied in scanning the structure and material properties, as well as predicting the thermal and mechanical characteristics of promising TBC materials for high and ultrahigh operating temperatures, which have been confirmed by subsequent experiments. Doping and alloying are also often employed in experiments to further lower thermal conductivities. Combining theoretical calculations and testing to identify next generation thermal insulator materials is already a standard procedure. A great number of prospective TBC materials have been developed in the last ten years by combining theoretical results with experiments. Anisotropic mechanical and thermal property prediction reveals an unique perspective on TBC material design. The next generation TBC advancements are anticipated to break through as a consequence of merging theoretical models, directed growth approaches, and solid solution design.

2.3. LITERATURE OF ATMOSPHERIC PLASMA SPRAY

Ozgurluk *et al* (2019) conducted research work on bond coating in which he researched that bond coatings are significant component of TBCs, and they may be produced by variety of methods, including Atmospheric Plasma Spray (APS), Vacuum Plasma Spray (VPS), High-Velocity Oxy-Fuel (HVOF), or Cold Gas Dynamic Spray (CGDS). The most essential aspect of TBC manufacture is the production of ceramic top coats. For this purpose, two approaches are often available: APS and EB-PVD. While he said that APS has a simple process and no substrate preheating is required, this is not the case with EB-PVD. As compared to EB-PVD, APS is cheaper, simpler, and faster production technology. When compared to Physical Vapor Deposition, the coating speed rate is also quicker.

Odhiambo *et al* (2019) established a detailed evaluation of porosity on air plasma-spray coatings. Optimal spraying settings were employed to obtain the best coatings with the least failures. Defects like pores may arise even with the optimum spraying settings. Various approaches of controlling pores in air plasma -sprayed coatings, with a specific focus on atmospheric plasma -chromium oxide coatings deposited. Techniques such as multilayered coatings, nanostructured

surface coating, doped with rare earth elements, laser surface re-melting, and a mixture of the above described techniques have been proposed to change pores.

Wang *et al* (2018) in this paper Atmospheric plasma spraying at various spraying powers was examined to develop nanostructured yttria stabilised zirconia (n-YSZ) thermal barrier coatings. According to the findings, these coatings had a bimodal microstructure composed of totally molten splats and partially melted nanostructured areas. The pores and nanostructure content of these coatings decreased as spraying power increased. The porosity of the coating reduced and hardness rose considerably after heat treatment at 1573 K compared to the as-sprayed layer, suggesting the sintering influence of the nanostructured coating. Nevertheless, after 24 hours of annealing at 1573 K, the porosity of the coating deposited with the lower spraying power remains over 10% demonstrating greater sintering resistance. As a conclusion, as spraying power was reduced, the YSZ coating included more nanostructure and pores, demonstrating improved adhesion strength but decreased micro hardness. The lower spraying power coating had a thermal cycling lifespan of more than 290 cycles at 1373 K, which was much longer than the others.

2.4. LITERATURE OF HOT CORROSION TEST

Goyal *et al* (2017) studied the effects of ZrO_2 inhibitor corrosion behaviour on elevated temperatures for Superfer 800H, Superni 75, and Superco 605 in a corrosion environment at 900 °C for 50 cycles. It was determined that ZrO_2 was capable of providing corrosion resistance to all alloys. It showed the highest resistance behaviour for Superni 75, and followed by Superco 605. It also offered some resistance to Superfer 800H. Detonation – gun spraying technique was used for coating process. He determined that the coating maintained constant contact with the substrate throughout the test, indicating that it had strong adhesive strength.

Ozgurluk *et al* (2019) also shown in his coating article that salt mixes of Na_2SO_4 and V_2O_5 were spread above surface of generated TBC specimens during the hot corrosion test. The test was performed on the samples at 1000°C (1273 K), with 4h intervals of cycling between furnace and ambient temp. These cycles were

repeated until the samples had degraded by about 40%. Following each 4hour session of testing at 1000°C, before being evaluated for cracking development, samples were given time to cool within the furnace. After that, the samples were applied with the $\text{Na}_2\text{SO}_4 + \text{V}_2\text{O}_5$ salt combination.

Vasudev *et al* (2020) investigated about the preparation and hot corrosion behavior of Inconel718-nano- Al_2O_3 based composite coatings with various nano- Al_2O_3 concentrations. The nano- Al_2O_3 powders were created using the hand granulation method. High-Velocity Oxy-Fuel method is used to deposit the composite coatings on the grey cast iron substrates. The corrosion behavior of naked and as-coated samples was investigated in a high-temperature furnace for 50 hours at 900°C. The oxidation mechanism of naked and coated specimens were determined using weight percentage data. The coating with the greatest powder concentration showed the higher hardness and resistance to corrosion behavior. In comparison to the GCI substrate, the inclusion of nano- Al_2O_3 and other protection oxides resulted in increased resistance of coatings.

Wang *et al* (2017) studied about the corrosion behaviour of nano structured zirconia in molten NaVO_3 salt environment. At 700 °C, the hot corrosion behaviour of both n-YSZ powders and their associated sodium metavanadate coatings was examined. During the test, NaVO_3 interacts with Y_2O_3 in YSZ to generate YVO_4 , resulting in a damaging phase transition of YSZ from tetragonal to monoclinic. The crystalline grain size of n-YSZ powders and their corresponding coatings had no effect on the corrosion processes. Yet, the coatings eroded faster than n-YSZ powders, and coatings formed from initial powders with crystalline grain sizes of 30nm exhibited better hot corrosion degradation resistance.

Muthu *et al* (2020) investigated and mentioned that due to the formation of a Cr_2O_3 protective layer, Ni-20%Cr coating has high corrosion resistance. The Cr_2O_3 layer has a increased melting point and is stable, allowing it to withstand intense degradation temperatures of up to 900 °C. The coatings were sprayed above the iron based superalloy by HVOF technique. Due to the formation of protective layer layers, both coatings provide increased resistance behaviour to spallation during the test in comparable turbine environments.

Fantozzi *et al* (2017) in his study tested the high temperature resistance to corrosion behaviour of several thermally sprayed Inconel 625 coatings at 550°C for 168 hours with a KCl salt layer. The purpose of the research was to determine the effect of chlorine corrosion on the complicated microstructure of thermally deposited coatings. All of the coatings performed well, by substrate damage and functioning as a protection against the corrosive environment. Where HVOF and TWAS coatings undergone the lowest corrosion degradation and HVOF sprayed coatings showed the highest corrosion degradation. The maximum temperature chlorine active oxidation corrosion procedure was applied to all of the coatings, resulting in the formation of porous and non-protective oxide and chromate layers on the surface. The corrosion products were primarily K_2CrO_4 and a mix of Cr, Ni, and Mo oxides. He also highlighted that the higher melting temperature of the particles during HVOF and TWAS spraying may have helped to reduce interconnected porosity and serve as a barrier for chlorine penetration.

Sadeghimeresht *et al* (2017) from his study the higher temperature corrosion resistance of HVOF thermal-sprayed NiCr and NiAl coatings subjected to 5 vol.% O_2 + 500 vppm HCl + N_2 with and without KCl salt deposit at 600 °C for up to 168 h was examined. The research was focussed at understanding the impact of corrosion of chlorine on the coating's complicated microstructure. He concludes that in the absence of KCl, both coatings worked effectively, they acted as a thermal barrier by regulating the substrate's corrosion against the chlorine-containing environment.

Ramkumar *et al* (2017) made an attempt to evaluate the influence of fillers on Inconel 625 and Inconel 718 by Tungsten Arc Welding. For combining 5mm thick plates of nickel based super alloys, two distinct Ni-Mo rich fillers, ERNiCrMo-4 and ERNiCrMo-10, were used. Hot corrosion tests were carried out on coupons that had been subjected to a molten salt environment of 60% Na_2SO_4 -40% V_2O_5 at 900°C. According to studies, both the parent metal and the fusion zone of ERNiCrMo-10 possessed higher corrosion resistance in elevated temperature environments. To analyze the hot corroded species, surface analytical methods were used.

Bijalwan *et al* (2019) in their study, Atmospheric plasma spraying was used to deposit iron based metallic glass composite coatings with a composition of Fe_{2.5}Cr_{6.7}Si_{2.5}B_{0.7}C (wt%) onto a mild steel base metal under ideal spraying conditions with regulated heat input variation. Nanoindentation was used to conduct a qualitative assessment for the influence of input heat on the alteration of the relative percentage of distinct stages, as a result the nanohardness of the coatings. Plasma sprayed coatings had minimal porosity, excellent hardness, and outstanding corrosion resistance, according to the study.

Sreenivasulu *et al* (2018) investigated and concluded that the air plasma spray method is widely used at numerous sectors because of the adaptability, lesser cost, and higher quality of the coatings produced. This current research analyses the high-temperature corrosion behaviour of Cr₃C₂-25NiCr and NiCrMoNb powder coatings sprayed by air plasma on base alloy 80A. The corrosion investigation was followed in an air and molten salt environment of molten salts for 50 cycles at 900°C. The corrosion behaviour of uncoated and coated alloy 80A was studied using thermogravimetric analysis. The conclusions show that the NiCrMoNb coating can be utilised as a protection coating in an air oxidation environment, and the Cr₃C₂-25NiCr coating may be employed in a molten salt environment.

Chellaganesh, Khan, and Jappes (2020) Hastelloy X alloy is chosen as a potential material for simulated oxidation and gas turbine hot corrosion at 1100°C in their study. The mass change per unit area in an oxidation experiment is gradual in a mixed salt environment and catastrophic in an atmospheric air environment. This finding might be related to the formation of oxide layers and their deformation after prolonged exposure. Electrical discharge machining (wEDM) is used to cut the samples to 10x10x3 mm dimensions. The rate of oxidation process in air is determined to be less than in a vanadium-containing mixed salt environment.

Sun *et al* (2019) in their research performed hot corrosion test at 900 °C on a modified aluminized NiCrAlYSi coating covered with mixed salt films of Na₂SO₄+K₂SO₄ and NaCl+Na₂SO₄. In terms of hot corrosion resistance in these two kinds of mixed salts, the modified aluminized NiCrAlYSi coating beat the aluminized NiCrAlYSi coating. According to results, the presence of Pt in aluminized NiCrAlYSi coating has helped to prevent the incidence of internal sulfidation and

degradation during the test. For both coatings, the interior oxide was Al_2O_3 and the interior sulphide was CrS. Certain Al-rich groups are produced with phase in both the coatings, which acts as an Al storehouse and also delivers ample Al during corrosion.

Moskal *et al* (2020) in this research under hot corrosion environment, the rapid degradation of a $\text{Sm}_2\text{Zr}_2\text{O}_7+8\text{YSZ}$ system in the form of Air Plasma Spraying Thermal Barrier Coatings of composites, double ceramic layer, and functionally graded system types were investigated. The noted pyrochlore phase breakdown was mostly attributable by interactions between $\text{Sm}_2\text{Zr}_2\text{O}_7$ and 8YSZ in line with the corroding mechanism characteristic of the corrosion process of nickel based superalloy. The degradation of the $\text{Sm}_2\text{Zr}_2\text{O}_7$ phase from a pyrochlore phase to a fluorite phase was the primary symptom of the disclosed process accelerated hot corrosion in liquid Na_2SO_4 deposits.

CHAPTER 3

AIM & SCOPE

3.1. AIM OF THE PROJECT

During combustion in aircraft engine, the vanes and blades of turbine will result in accelerated oxidation or sulfidation due to reaction with sodium sulphate and vanadium (V) oxide that form from fuel. The erosion and lifetime can be increased by means of selecting appropriate coating technology and coating composition. In this work oxidation stability and corrosion behaviour of Samarium Zirconate mixed with YSZ coated on Hastelloy X is studied.

3.2. SCOPE

In this work, the main aim is to increase the resistance of hot corrosion behaviour of the coated specimens. In this regard the multilayered TBC is used to extend the corrosion resistance. The pure 7YSZ is most commonly used TBC composition in aviation applicants. Instead 7YSZ doped with Samarium Zirconate ($\text{Sm}_2\text{Zr}_2\text{O}_7$).

CHAPTER 4

MATERIALS AND METHODS

4.1. MATERIAL

Nickel based alloy i.e., Hastelloy X with a thickness of 5mm is used as the substrate which mostly used in elevated temperature in aircraft turbine applications. We have done Atmospheric plasma (TBC) spraying on the substrate by using 7YSZ mixed with Samarium Zirconate as a thermal coating material. Hastelloy X is a nickel-based super alloy with high resistance and durability to degradation up to 2200°F.

4.2 METHODOLOGY OF COATING

Hastelloy X is selected as the base material, as it can withstand better at higher temperatures. Then the pre-processing like sample preparation, surface cleaning, surface activation and work is performed. After the pre-processing method, coating process is performed. The process is performed by Atmospheric Plasma Spray process. Firstly, bond coat is coated over the base material. Next the coating composition process is performed. Here above the bond coat the over coat is coated by Atmospheric Plasma Spray process. Three different types of coatings are followed and the specimens are subjected to hot corrosion test. Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) results of the specimens are collected 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.

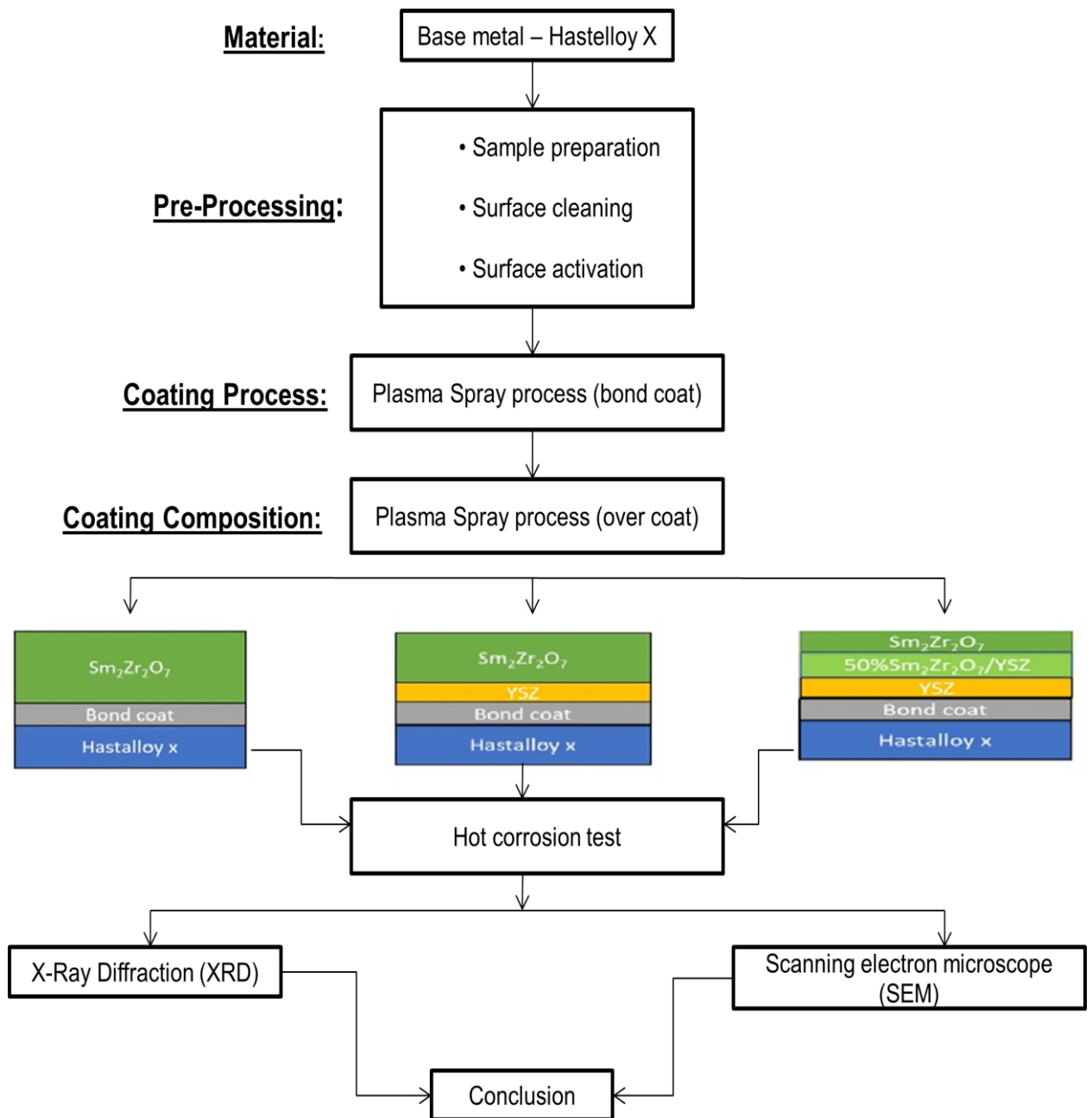


Fig4.1: Flow chart representing methodology of coating

4.3 COATING THICKNESS

4.3.1. Single layer - Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$

Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$ as the TBC material with a thickness of $250\mu\text{m}$ along with bond coat of thickness $50\mu\text{m}$ is coated above the substrate. The microstructure of substrate before and after coating were explained in the result and discussion chapter. The as sprayed pure $\text{Sm}_2\text{Zr}_2\text{O}_7$ microstructure is illustrated in the figure 5.2. The tested $\text{Sm}_2\text{Zr}_2\text{O}_7$ after subjecting to the hot corrosion test.

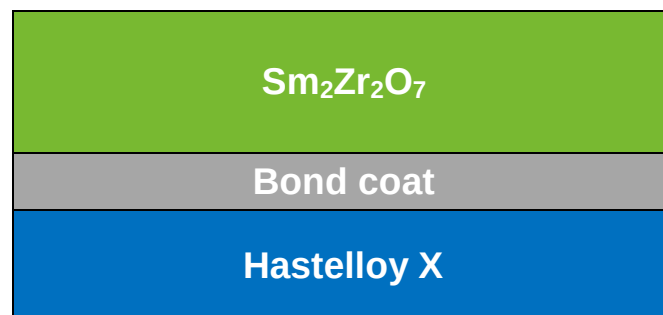


Fig4.2: Single layered coating – Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$

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

The base metal Hastelloy X is bond coated with $50\mu\text{m}$ thickness. Above the bond coat the double layered sandwich form of coating is done with 7YSZ is coated with a thickness of $100\mu\text{m}$ and above the 7YSZ coating pure $\text{Sm}_2\text{Zr}_2\text{O}_7$ is coated with a thickness of $150\mu\text{m}$.

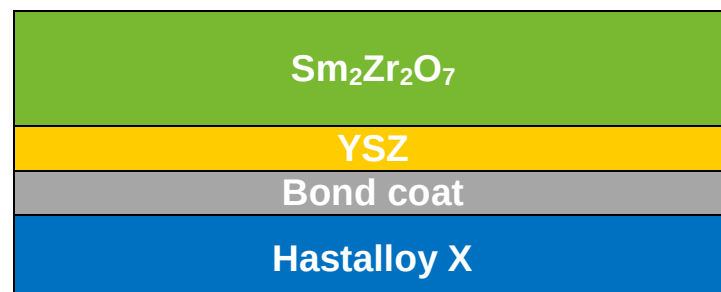


Fig4.3: Double layered coating – YSZ / Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$

4.3.3. Composite Multilayer – 7YSZ / 50% $\text{Sm}_2\text{Zr}_2\text{O}_7$ + 50% 7YSZ / Pure

$\text{Sm}_2\text{Zr}_2\text{O}_7$

The base metal Hastelloy X is bond coated with 50 μm thickness. Above the bond coat the multi layered sandwich form of coating is done with 7YSZ is coated with a thickness of 50 μm and above the 7YSZ coating 50% $\text{Sm}_2\text{Zr}_2\text{O}_7$ + 50% 7YSZ is coated with thickness of 100 μm and above that pure $\text{Sm}_2\text{Zr}_2\text{O}_7$ is coated with thickness of 100 μm .

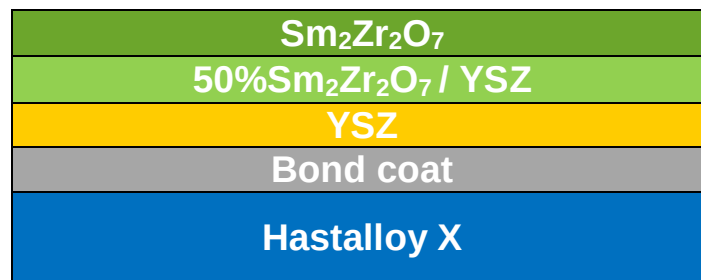


Fig4.4: Multi layered coating – 7YSZ / 50% $\text{Sm}_2\text{Zr}_2\text{O}_7$ + 50% 7YSZ / Pure $\text{Sm}_2\text{Zr}_2\text{O}_7$

4.4. MATERIAL PREPARATION FOR PLASMA SPRAY COATING PROCESS

Material shaping and sizing in accordance with coating requirements. The gun in the plasma spraying process should use the least amount of powder to conduct the coating operation (even the coating surface needs small quantity of powder, the spraying will be done only if we fill the gun at its minimum requirement level). Similarly, when it comes to specimen size, the specimen should be able to be held by machine holders during the spraying process. Little specimens, such as 10x10mm are difficult to handle. As a result, specimen size should be done appropriately.

The 300 μm thickness coating is applied to the substrate utilising the APS technique, which includes bond coat and TBC. As stated in the coating methods section, 3 types of coating are applied to the substrate, each having a distinct composition. The Microscopic test findings clearly show the differences between the 3 types of coatings.

4.5. MATERIAL PREPARATION FOR TESTING

4.5.1. SEM (Scanning Electron Microscope)

Originally, the specimens were shaped according to the testing criteria, such as 15x15mm size, and polished with an emery sheet with a grade starting from (100, 400, 800, 1000, 1200, 1500, and 2000) for better results during microscopy. During scanning the substrate, the roughness from the surface of the substrate will be eroded and an even surface will be achieved by polishing from low to fine grade. The findings were discussed in the result and discussion chapter.

4.5.2. XRD (X-Ray Diffraction)

The method of X-ray diffraction (XRD) is followed to determine the content present and crystalline structure of a material. This pattern may be used to determine the crystal's atomic structure, including the spacing and arrangement of atoms inside it. XRD is often used for crystalline material phase identification and can offer information on unit cell size. It is most effective for bigger crystals such as macromolecules and inorganic compounds, but it may also be used for smaller crystals to assess sample composition, crystallinity, and phase purity.



Fig4.5: XRD Equipment

4.5.3. Hot Corrosion

Based on the testing facility's requirements, the samples were fashioned in a circular fashion, with a diameter of 20. Next a 10mg/cm² combination of 45% of sodium sulphate (1.8mg) and 55% of vanadium (V) oxide (2.2mg), as well as a small amount of distilled water, is added. The alumina boats are used to transport specimens into the furnace. As an example, an alumina boat can resist temperatures of more than 1000°C. The specimens after applying the salts placed in the alumina boat can be seen from the figure 4.4. A muffle furnace was used to conduct the test. This mixture is then placed in a Hot air oven at 110 °C for some time for the samples to be dry. Fig 4.5 represents the hot air oven. Following that, hot corrosion tests were carried out. The samples were held inside the muffle furnace for a temperature of 1000 °C for 50 hours, 8 hour once the samples being removed to check for cracks or deformation of the coatings. Fig 4.6 represents the muffle furnace. The coatings deformed after 50 hours, and the test was finished. After the hot corrosion test, samples were collected for XRD and SEM analysis. XRD determines whether a sample is amorphous or crystalline. SEM photos are acquired to learn about the surface composition and topography of a substance. The XRD and SEM data are presented in the results and discussion.



Fig4.6: specimens in alumina boat



Fig4.7: Hot air oven



Fig4.8: Muffle furnace

The specimen surface morphology pictures taken from the scanning electron microscope after completing the corrosion test were compared in the result and discussion section, and the changes that happened are thoroughly discussed below. The alterations that happened in one of the specimens, namely the composite multilayer coating, had a positive result. After 50 hours of testing at 1000°C, the single layer coating substrate entirely peeled off by applying the salts sodium sulphate and vanadium (V) oxide. Fig 4.7 shows the comparison between the ascoated samples and the samples after hot corrosion test.

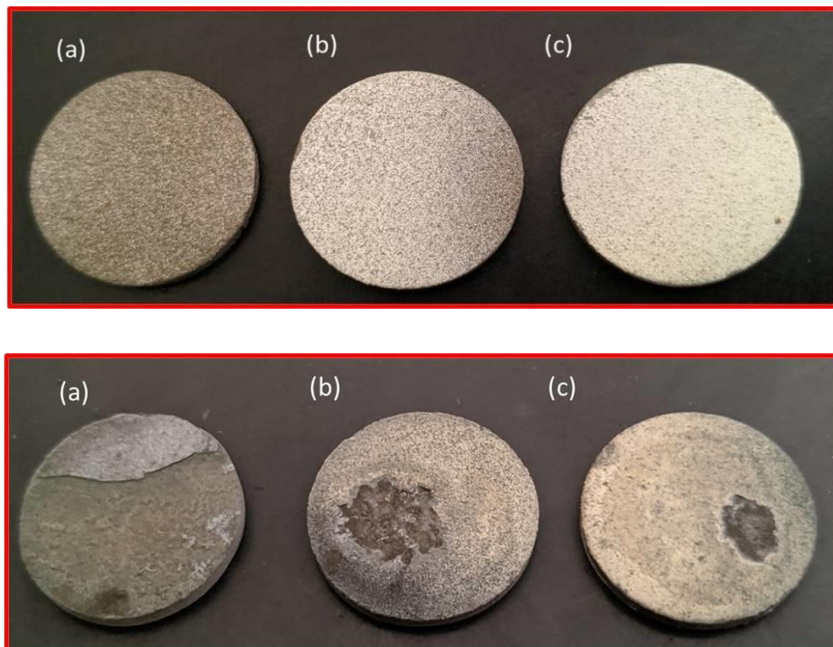


Fig 4.9: Comparison of samples before and after hot corrosion test

CHAPTER 5

RESULT AND DISCUSSION

5.1. SURFACE MORPHOLOGY OF THREE SAMPLES

The illustration depicts a cross-sectional view of the 3 as coated samples. Figure 5.1 shows the evident creation of the Bond coat and TBC layer.

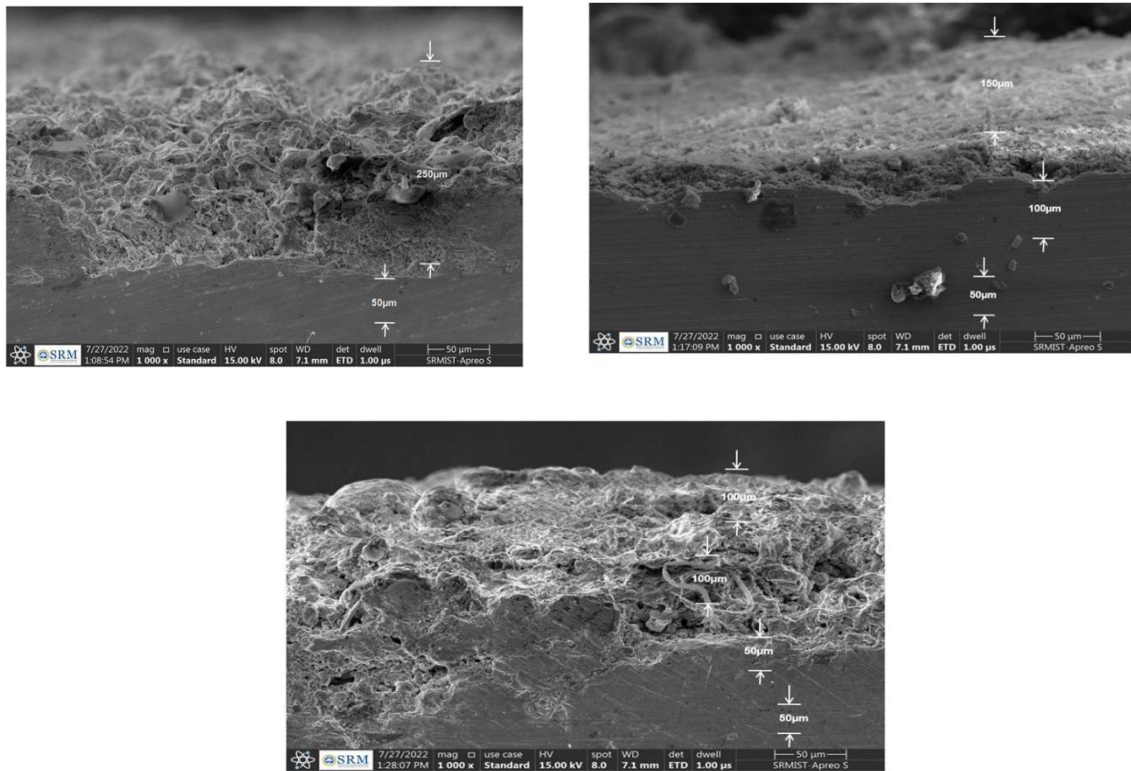


Fig 5.1: Optical images of cross – sectional view of substrate

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. In addition, Figure 5.2 depicts the comparison between surface morphology of ascoated samples prior to testing and surface morphology of Heat treated samples.

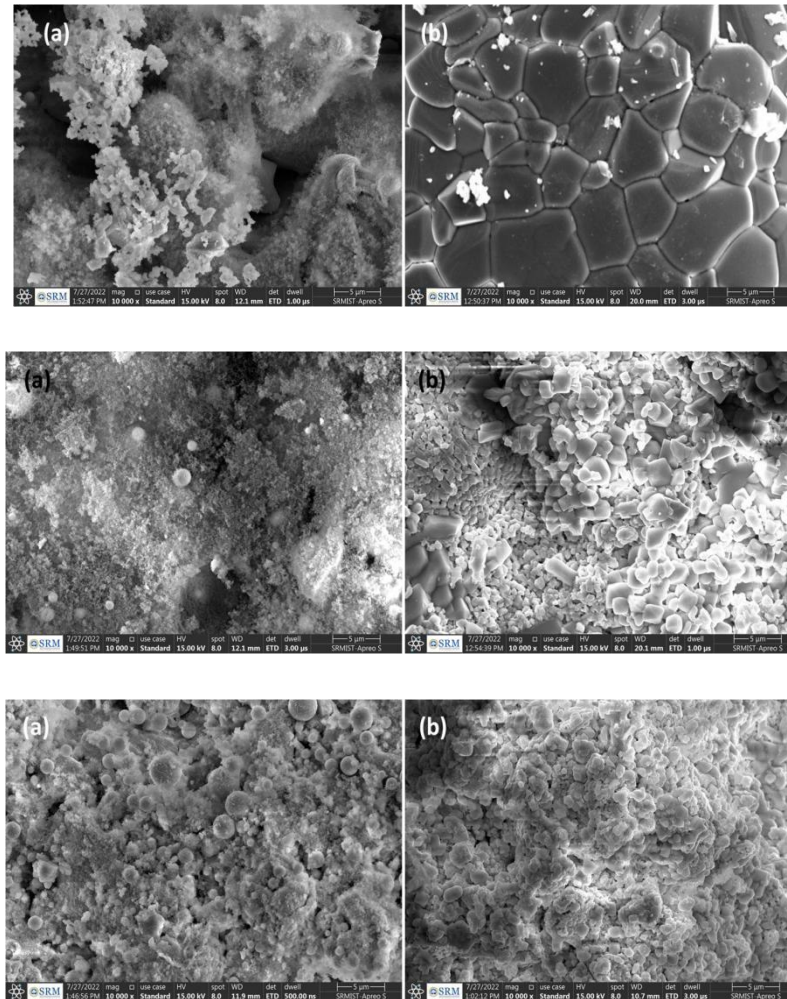


Fig 5.2: Comparison of Surface morphology of Ascoated samples and heat treated samples

a) Surface morphology of ascoated samples

b) Surface morphology of specimens after hot corrosion

The figure depicts the specimens after 50 hours of being subjected to the hot corrosion 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 hot corrosion.

5.2. SURFACE MORPHOLOGY AND STUDY AFTER TEST

Figure 5.2 shows that the surface microstructural picture prior to hot corrosion does not include many pores and cracks. For the three distinct kinds of coatings, the surface morphology after test shows a rise in salt content and varying separation of grains. There are more cracks and pores in the pure $\text{Sm}_2\text{Zr}_2\text{O}_7$ layer or single layer. The creation of cracks may be noted in the Fig 5.2. The salts have a significant influence on the double layer TBC. The uneven development of salts may be picked out from the following images. The presence of increased weight % of sodium is detected in the double layer and vanadium in composite multilayer.

5.2.1. XRD (X-Ray Diffraction) of single layer coating

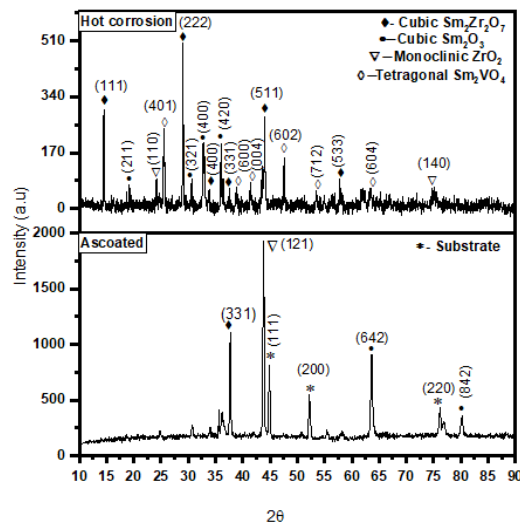


Fig 5.3: XRD representation of single layer

As per the XRD results, it showed the presence of cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$, Cubic Sm_2O_3 , Tetragonal YVO_4 and Monoclinic ZrO_2 . Tetragonal YVO_4 is formed by the reaction between yttrium and V_2O_5 . During test the samarium zirconate coating and the molten salts reacted with each other and has formed Monoclinic ZrO_2 . The pyrochlore $\text{Sm}_2\text{Zr}_2\text{O}_7$ will lose some molecules and break into cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$. 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 .

5.2.1.1. Microstructural analysis of single layer coating

Figure 5.4 depicts the porosity and fractures generated in the single layer substrate coating, as well as a scattered structure representing the bond coat. The salt content is notably more substantial compared to the other types of coatings.

Table 5.1: Elements in single layer

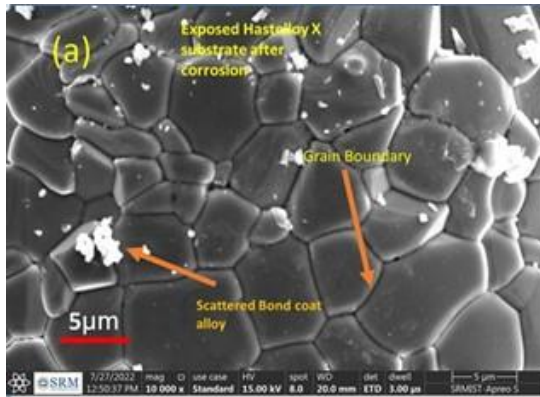


Fig 5.4: Surface image of single layer

Element	Weight%
Samarium	25.58
Zirconium	40.91
Oxygen	33.51
Total	100

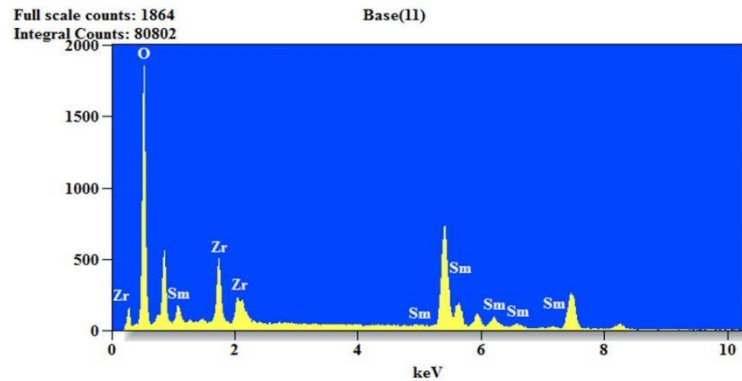


Fig 5.5: Composition in single layer

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 SZ1 or single layer sample the element constitution present is arranged in the above table. By viewing the SEM images of the Ascoated sample and Hot corrosion tested sample we can visualize changes in the structure and oxide formation due to the presence of Na_2SO_4 and V_2O_5 salts and severe oxidation the metal after testing. The coating is peeled off and corrosion mechanism is very severe.

5.2.2. XRD of double layer coating

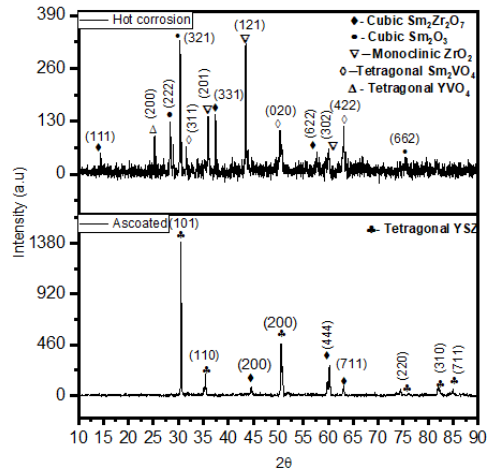


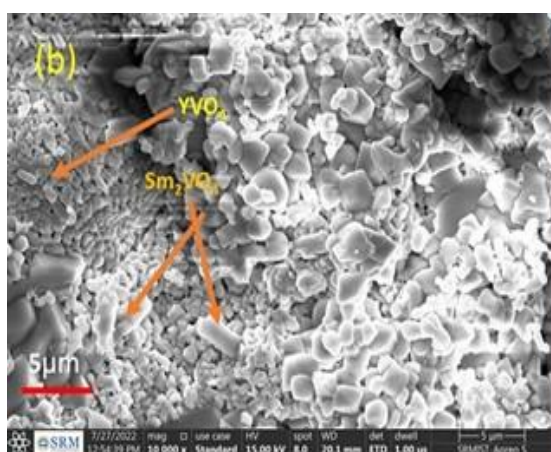
Fig 5.6: XRD representation of double layer

As per the XRD results, it showed the presence of cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$, Cubic Sm_2O_3 , Tetragonal YVO_4 , Monoclinic ZrO_2 and Tetragonal Sm_2VO_4 , and Tetragonal YSZ is present in substrate material. Tetragonal YVO_4 is formed by the reaction between yttrium and V_2O_5 . During the test the samarium zirconate coating and the molten salts reacted with each other and has formed Monoclinic ZrO_2 . The pyrochlore $\text{Sm}_2\text{Zr}_2\text{O}_7$ will lose some molecules and break into cubic $\text{Sm}_2\text{Zr}_2\text{O}_7$. 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 .

5.2.2.1. Microstructural analysis of double layer coating

Figure 5.7 depicts the porosity and fractures generated in the double layer substrate coating, as well as rod like structure representing the salts. When compared to single layer coating, the salt concentration is noticeably lower.

Table 5.2: Elements in double layer



Element	Weight%
Samarium	25.36
Zirconium	44.34
Oxygen	30.30
Total	100

Fig 5.7: Surface image of double layer

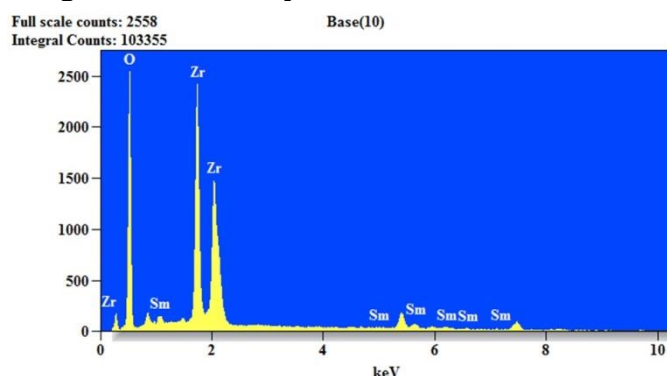


Fig 5.8: Composition in double layer

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 viewing the SEM images of the Ascoated sample and Hot corrosion tested sample we can visualize changes in the structure and oxide formation due to the presence of Na_2SO_4 and V_2O_5 salts and severe oxidation the metal after testing. The coating is damaged and but corrosion mechanism is less compared to SZ 1. From the Fig 5.7 we can clearly confirm the presence of tetragonal YVO_4 and tetragonal Sm_2VO_4 . But we can say that the double layer performed resisted well at higher temperatures when comparing to the single layer.

5.2.3. XRD of composite multilayer coating

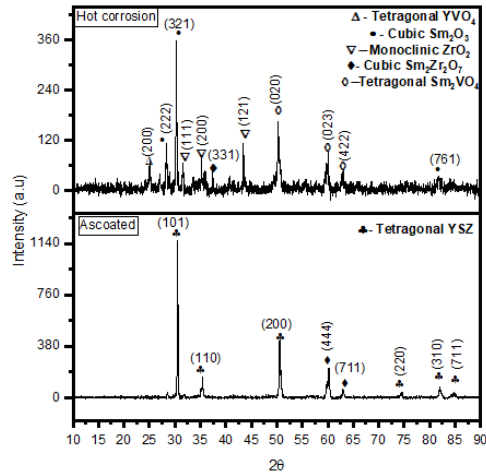


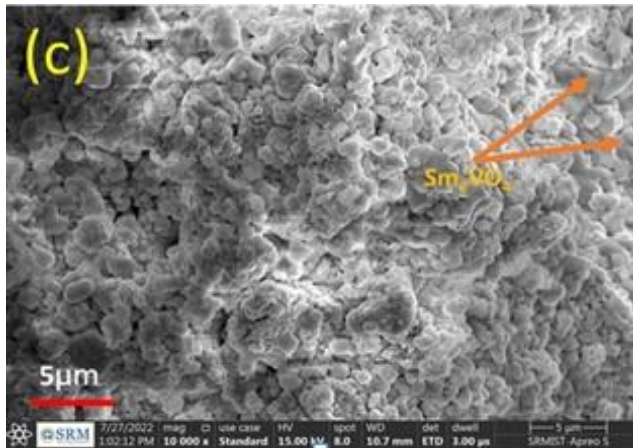
Fig 5.9: XRD representation of composite multilayer

As per the XRD results, it showed the presence of cubic Sm₂Zr₂O₇, Cubic Sm₂O₃, Tetragonal YVO₄, Monoclinic ZrO₂, Tetragonal Sm₂VO₄ and Tetragonal YSZ is present in substrate material. Tetragonal YVO₄ is formed by the reaction between yttrium and V₂O₅. During the test the samarium zirconate coating and the molten salts reacted with each other and has formed Monoclinic ZrO₂. The pyrochlore Sm₂Zr₂O₇ will lose some molecules and break into cubic Sm₂Zr₂O₇. The separated Sm₂ molecules from Sm₂Zr₂O₇ will react with Oxygen and forms cubic Sm₂O₃.

5.2.3.1. Microstructural analysis of composite multilayer coating

Figure 5.10 depicts the porosity and fractures generated in the composite multilayer substrate coating, as well as rod like structure representing the salts. When compared to other two coatings, the salt concentration is noticeably lower.

Table 5.3: Elements in composite multilayer



Element	Weight%
Samarium	28.59
Zirconium	44.45
Oxygen	26.96
Total	100

Fig 5.10: surface image of composite multilayer

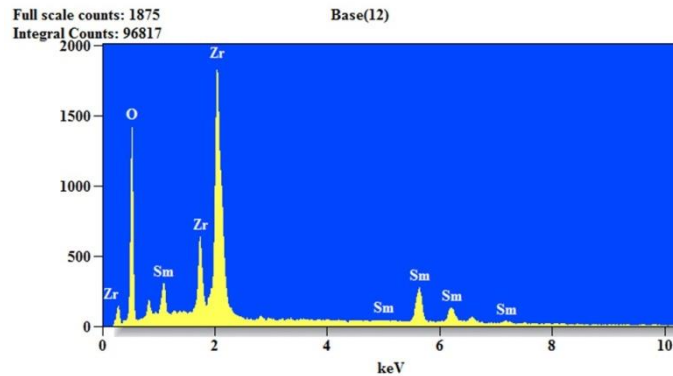


Fig 5.11: Composition in composite multilayer

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 composite multilayer sample the element constitution present is arranged in the above table. By viewing the SEM images of the Ascoated sample and hot corrosion tested sample we can visualize changes in the structure and oxide formation due to the presence of Na_2SO_4 and V_2O_5 salts and severe oxidation the metal after testing. The coating is damaged and but corrosion mechanism is less compared to the other two coatings.

5.2.4. Hot corrosion test analysis

The specimens were placed in a higher temperature of 1000°C for 8 hours before being sent to SEM. After that subjecting the identical specimens to the same extreme temperature for 50 hours, we discovered that the whole TBC layer had been destroyed and peeled off from the single layer coated substrate. From the Fig 5.12 we conclude that the TBC coated layer was not damaged by the peeling behaviour of the single layer coated sample.

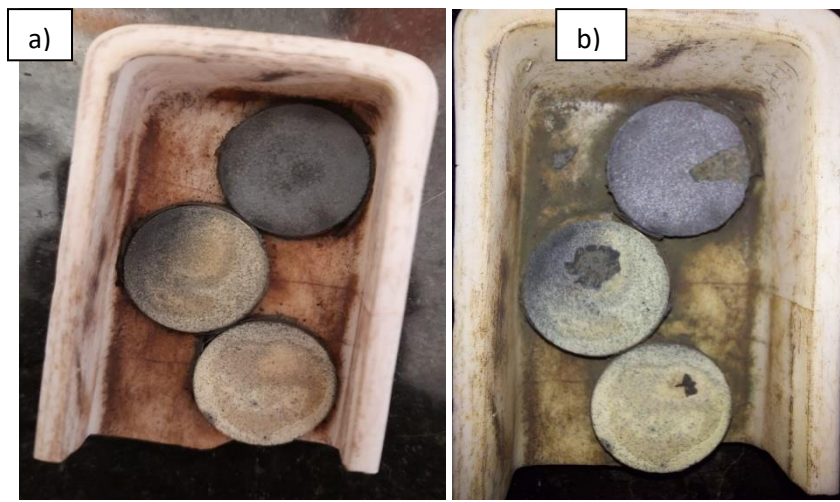


Fig 5.12 Surface images of specimens after exposing to hot corrosion effect

a) After 5 hours at 1000°C

b) After 35 hours at 1000°C



Fig 5.13 Damage of TBC without damage to substrate

CHAPTER 6

SUMMARY AND CONCLUSION

6.1. SUMMARY

Hastelloy 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 Hastelloy X is exposed to those salts at that high temperature corrosion will occur. As a conclusion, TBC is the most often utilised resistivity-increasing method, and YSZ, with 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 7 weight % yttrium stabilised zirconia mixed with two coatings types provided insight into the effect of molten salts where the specimens undergone hot corrosion effect at enhanced temperature of about 1000°C .

To test the TBC's resistance to salts at extreme temperatures, a 10mg/cm² combination of 1.8mg of sodium sulphate and 2.2mg of vanadium (V) oxide was applied to the TBC layer and exposed to 1000°C temperature for 5 hours, with changes in surface morphology observed by SEM. The specimens were then treated to the same temperature for 50 hours, and peeling of the TBC layer occurred in all reactions with the thermally generated oxide layer between the bond coat and TBC layer.

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 method. The specimens were then subjected to a molten salt environment containing sodium sulphate and vanadium (V) oxide. As a result of TGO (thermally grown oxide) contact with TBC composition, the TBC surface alone broke off from all specimens. Yet, when vanadium and sodium sulphate are evenly distributed throughout the surface of without leaving the edges, the specimens may withstand the coating for up to 50 hours at 1000°C. It was discovered that,

1. The single layer after subjected to hot corrosion effect it lead to formation of cracks and bi-products, TBC layer completely peeled off at 50hours and no damage occurred to the substrate material. The comparison of before and after test image can be noticed in the Fig 4.7. Also can notice the scattered bond coat in Fig 5.4.
2. The double layer coating from the SEM results as in Fig 5.7 showed the presence of tetragonal Sm_2VO_4 and tetragonal YVO_4 . EDS results as in Fig 5.8 and Table 5.2 shows the presence of Zirconium in higher percentage. And the small cylindrical tube like structures formed in the surface morphology shown in Fig 5.7 represents the composition of vanadium, samarium and Zirconium.
3. The composite multilayer coating showed lesser reaction with vanadium and lesser cylindrical tube like structures are formed when compared to double layer coating after subjected to test by exposing to molten salts. TBC layer slightly peeled off with no significant damage as shown in Fig 4.7. From the Fig 5.10 we can clearly notice the formation of tetragonal Sm_2VO_4 . Composite multilayer coating performed very well comparing to the other two coatings. We conclude that using of composite multilayer coating provides best result to high temperature corrosion behaviour in presence of molten salts.

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