

Evaluation of hot corrosion behavior on Yb_2O_3 doped $\text{Gd}_2\text{Zr}_2\text{O}_7$ / YSZ Bilayer thermal barrier coatings on hastelloy x

Submitted in partial fulfilment of the requirements for the award of Bachelor of
Engineering / Technology degree in B.E Mechanical Engineering

Project Carried Out By

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DEPARTMENT OF MECHANICAL ENGINEERING

SCHOOL OF MECHANICAL ENGINEERING

**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 **SUDARSAN JP (38150114)**, **STEPHANAS ABHISHEK L(38150113)** who carried out the project entitled "EVALUATION OF HOT CORROSION BEHAVIOUR ON YB_2O_3 DOPED $\text{Gd}_2\text{Zr}_2\text{O}_7$ / YSZ BI-LAYER THERMAL BARRIER COATINGS ON HASTELLOY X" under our supervision from **August 21 to March 22**.

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Submitted for Viva voce Examination held on _____

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External Examiner

DECLARATION

We , **SUDARSAN JP (38150114)** and **STEPHANAS ABHISHEK L (38150113)** , hereby declare that the project Report entitled “**Evaluation of hot corrosion behavior on Yb₂O₃ doped Gd₂Zr₂O₇ / YSZ Bilayer thermal barrier coatings on hastelloy x**” done by us under the guidance of **Dr.A.Anderson ME, (Ph.D)** (Internal) at sathyabama Institute of Science and Technology , Jeppiar nagar ,Chennai – 600119 is submitted in partial fulfilment of the requirement for the award Bachelor of Engineering degree in Mechanical Engineering .

DATE:

PLACE:

SIGNATURE OF THE CANDIDATES

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CHAPTER-1

INTRODUCTION

The demand for improved performance in high-temperature mechanical systems has led to increasingly harsh operating environments, particularly for the components in advanced gas turbine engines. Future improvements in gas turbine performance will require even higher operating efficiencies, longer operating lifetimes, and reduced emissions. Achieving these requirements will necessitate still further increases in the gas inlet temperatures and, consequently, the development of structural materials inherently capable of higher temperature performance. Advanced cooling schemes coupled with thermal barrier coatings (TBCs) can enable the current families of superalloy components to meet the materials needs for the engines of tomorrow. Thermal barrier coating systems are currently capable of providing metal temperature reductions of up to about 140°C, whereas potential benefits are estimated to be greater than 170°C. The use of TBCs in combination with cooling schemes has enabled the operation of gas turbine engines having combustion gas temperatures in excess of 250°C above the melting temperature of the superalloy component (e.g., early stage turbine blades and vanes). Moreover, the use of TBCs at lower operating temperatures reduces the metal temperature, which in turn increases the service life of the component. In fact, current engine design primarily relies on the lifetime extension benefits of TBCs and does not fully exploit the significant performance improvements TBCs are capable of providing. This is because current, state-of-the-art TBCs are not considered “prime reliant” to the extent that they can be used with assurance that failure will not occur

This limitation derives from a number of issues, including the following:

- TBCs do not provide self-renewing protection. When they spall the thermal protection is lost.
- TBC service life is subject to scatter that to date has not been adequately taken into account.

- There are currently no effective methods (e.g., nondestructive evaluation) that can act as quality control and/or monitor for TBCs.
- Life prediction for TBCs is not as accurate as is needed.

1.1 USES OF THERMAL BARRIER COATING

Thermal barrier ceramic coatings are becoming more common in automotive applications. They are specifically designed to reduce heat loss from engine exhaust system components including exhaust manifolds, turbocharger casings, exhaust headers, downpipes and tailpipes. This process is also known as "exhaust heat management". When used under-bonnet, these have the positive effect of reducing engine bay temperatures, therefore reducing the intake air temperature.

Although most ceramic coatings are applied to metallic parts directly related to the engine exhaust system, technological advances now allow thermal barrier coatings to be applied via plasma spray onto composite materials. It is now commonplace to find ceramic-coated components in modern engines and on high-performance components in race series such as Formula 1. As well as providing thermal protection, these coatings are also used to prevent physical degradation of the composite material due to friction.

This is possible because the ceramic material bonds with the composite (instead of merely sticking on the surface with paint), thereby forming a tough coating that doesn't chip or flake easily.

Although thermal barrier coatings have been applied to the insides of exhaust system components, problems have been encountered because of the difficulty in preparing the internal surface prior to coating.

1.2. MATERIALS EXPLANATION

1.2.1 YSZ

YSZ is the most widely studied and used TBC because it provides excellent performance in applications such as diesel engines and gas turbines.

Additionally, it was one of the few refractory oxides that could be deposited as thick films using the then-known technology of plasma spraying.[2] As for properties, it has low thermal conductivity, high thermal expansion coefficient, and low thermal shock resistance. However, it has a fairly low operating limit of 1200°C due to phase instability, and can corrode due to its oxygen transparency.

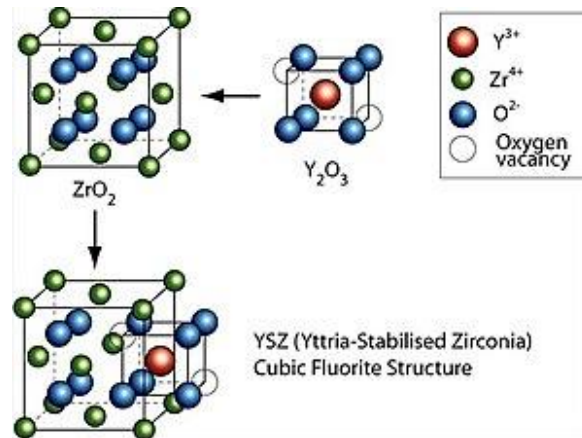


Fig 1.1: yttria –stabilised zirconia

1.2.2 Hastelloy

Hastelloy is a corrosion-resistant nickel alloy that contains other chemical elements such as chromium and molybdenum. This material has high temperature resistance and exceptional corrosion resistance. Hastelloy is a term used to describe nickel metal that has been alloyed to increase corrosion resistance. This is primarily done through the addition of molybdenum and chromium. There are many grades of Hastelloy, with each grade having a different chemical makeup to be optimized for a specific set of properties.

With Hastelloy typically containing high quantities of both molybdenum and chromium, it performs better in severely corrosive environments than other specialty metal products such as Incoloy. The addition of molybdenum also increases the general workability. Hastelloy alloys are also ductile and are easily fabricated and formed.



Fig 1.2 : hastelloy

1.2.3 Nimonic

Nimonic is a registered trademark of Special Metals Corporation that refers to a family of nickel-based high-temperature low creep superalloys. Nimonic alloys typically consist of more than 50% nickel and 20% chromium with additives such as titanium and aluminium.

The main use is in gas turbine components and extremely high performance reciprocating internal combustion engines. The Nimonic family of alloys was first developed in the 1940s by research teams at the Wiggin Works in Hereford, England, in support of the development of the Whittle jet engine.

1.2.4 Inconel

Inconel is a registered trademark of Special Metals Corporation for a family of austenitic nickel-chromium-based superalloys. Inconel alloys are oxidation-corrosion-resistant materials well suited for service in extreme environments subjected to pressure and heat

1.2.5 Nickel Alloys

Nickel alloys are popular in aerospace industry to make turbine blades, discs and other critical jet engine parts because they provide excellent adhesion, corrosion protection, hardness, wear and erosion resistance, is appropriate for applications where stress needs to be minimized and are structurally tough and have fantastic creep resistance properties. In aircraft engines, which operate at very high temperatures and stresses, special nickel-based alloys are used owing to their ability to retain their strength at elevated temperatures. In addition to this, nickel alloys are used in the exhaust valves, thermostat rods, tanks and piping for liquefied gas storage.



Fig 1.3 :Nickel alloy

1.3 DOPPING

In semiconductor production, doping is the intentional introduction of impurities into an intrinsic semiconductor for the purpose of modulating its electrical, optical and structural properties. The doped material is referred to as an extrinsic semiconductor.

1.4 COATING

Thermal Barrier Coating (TBC) is a coating produced by a process in which molten or softened particles are applied to impact onto a substrate. A common feature of all thermal spray coatings is their lenticular grain structure resulting from the rapid solidification of small globules, flattened from striking a cold surface at high velocities. Modern TBCs are required not only to limit heat transfer through the coating but also to protect engine components from oxidation and hot corrosion. By attaching an adherent layer of a low thermal conductivity material to the surface of an internally cooled gas turbine blade, a temperature drop can be induced across the thickness of the layer. This results in reduction of the component surface temperature to which it is applied. This temperature drop reduces the oxidation rate of the bond coat applied to metal components, and so delays failure by oxidation. It also retards the onset of thermally induced failure mechanisms (i.e., thermal fatigue) that contribute to component durability and life. It is important to note that coatings of this type are currently used only for component life extension at current operating temperatures. They are not used to increase the operating temperature of the engine. However, the development of a TBC system, for which the probability of failure is sufficiently low, would allow these coatings to be used to increase the engine operating temperature and lead to significant improvements in engine performance. The first TBCs, applied in the 1970s, were aluminide coatings. Improved ceramic coatings became available in the 1980s. These coatings increase turbine blade capability. The coatings also improve blade life by almost doubling the life of turbine blades in some cases. Thermal spraying is a process in which molten, semi-molten or solid particles are deposited on a substrate. 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. The classification of spray techniques is based on the way of generation of such streams. They are Arc Wire thermal spray process, Detonation spray process, High Velocity Oxy Fuel process, Cold Spray coating process, Plasma spray process, ESVAD, EBPVD, This research used plasma spray coating for the 7 coating process. Among all above coating technologies, plasma spraying process is the most widely used technique for the coating purpose

COATING MATERIALS USED FOR HIGH TEMPERATURE

The primary purposes of high-temperature structural coatings are to enable high temperature components to operate at even higher temperatures, to improve component durability, and to allow use of a broader variety of fuels in land-based and marine-based engines. Although high-temperature coatings protect the substrate, the demarcation between coating and substrate (either metal or nonmetal) is becoming increasingly blurred. Thermal barrier coating (TBCs) which are often based on yttria-stabilized zirconia (YSZ) can reduce the surface temperature up to 200°C.

a)ZIRCONIUM OXIDE

Zirconium dioxide (ZrO_2), sometimes known as zirconia (not to be confused with zircon), is a white crystalline oxide of zirconium. Its most naturally occurring form, with a monoclinic crystalline structure, is the mineral baddeleyite. A dopant stabilized cubic structured zirconia, cubic zirconia, is synthesized in various colours for use as a gemstone and a diamond simulant.



Fig 1.4 Zirconium oxide

Zirconia is chemically unreactive. It is slowly attacked by concentrated hydrofluoric acid and sulfuric acid. When heated with carbon, it converts to zirconium carbide.

When heated with carbon in the presence of chlorine, it converts to zirconium tetrachloride. This conversion is the basis for the purification of zirconium metal and is analogous to the Kroll process

b) YTTRIUM OXIDE

In the recent years scientists have intensified their research in the field of nanoparticles mainly due to the innovative and unexpected results that are achieved through altering of the atomic and molecular properties of various elements. Nanoparticles have found their uses in the field of bio technology, drug delivery, optics and electronics.



Fig 1.5 Yttrium oxide

Yttrium oxide (Y_2O_3) nanoparticle is an air-stable, solid substance white in color. It is used in the field of material sciences, to make phosphors that are used in imparting the red color of the picture tubes in televisions. Another major use of the yttrium oxide nanoparticles is in inorganic synthesis.

1.4.1 Plasma spray:

Uses a plasma-forming gas (typically argon or nitrogen) as both the heat source and propelling agent for the coating material. A high voltage arc struck between an anode and cathode within the gun ionizes the gas, which is then forced through a convergent/divergent nozzle.

As the gas exits the nozzle, it reverts to its natural state, liberating large amounts of heat. Powdered coating material is injected into the hot gas stream, where it is melted and projected at high velocity onto a prepared substrate.

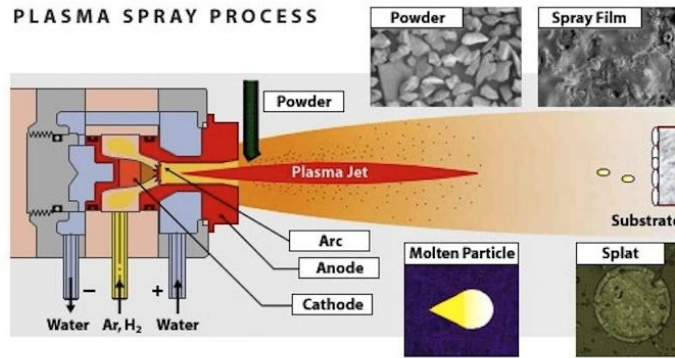


Fig 1.6 :Plasma spray

1.4.2 Wire flame spray

Applies coatings of any metal that can be drawn into a wire. It has a melting point below that of a combustion flame. Drive rolls feed the wire through a flame-spray gun to its nozzle where the wire is melted in an oxygen/fuel-gas (oxy fuel) flame after which compressed air atomizes the metal and projects it onto the prepared surface

Advantages: Best choice for all-purpose spraying. Coatings can be applied rapidly and at low cost.

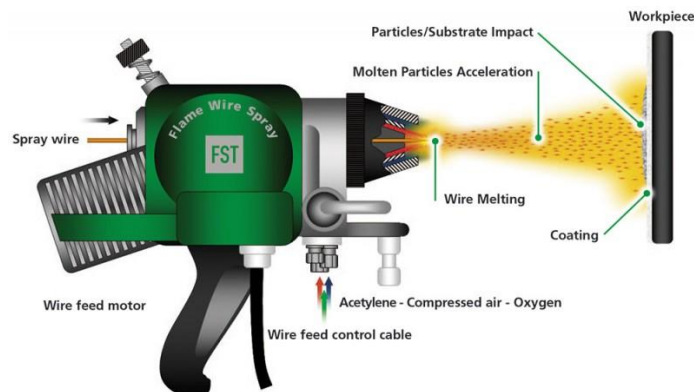


Fig 1.7 :Wire flame spray

1.4.3 Electric-arc spray

It applies metal coatings in wire form. Push/pull motors feed two electrically charged wires through the spray gun. When their tips touch at the gun head, an arc is created which melts the wires at temperatures above 3,980 °C after which compressed air atomizes the molten metal and projects it onto a prepared surface.

Advantages: It can spray up to a large area and can also apply thick coatings. Its built-in flexibility permits tailoring of coating characteristics, such as hardness and surface texture

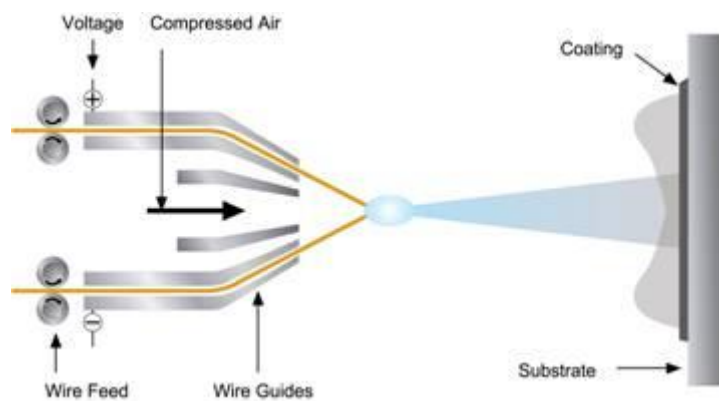


Fig 1.8:Electric arc spray

1.4.4 High-Velocity Oxy-fuel (HVOF)

It uses high-velocity combustion of mixtures of oxygen and a fuel gas to heat and propel semi- molten-to-molten powdered materials. The sprayed particles impact the substrate with extremely high kinetic energy and with negligible oxidation of the coating material. Advantages: The HVOF method produces coatings that have the highest bond strength and lowest porosity. Coatings also have low internal stresses, which allow thick layers to be deposited, and fine as sprayed surface finishes. HVOF is well suited to producing high-quality tungsten and chromium-carbide coatings.

High Velocity Oxy-Fuel Process

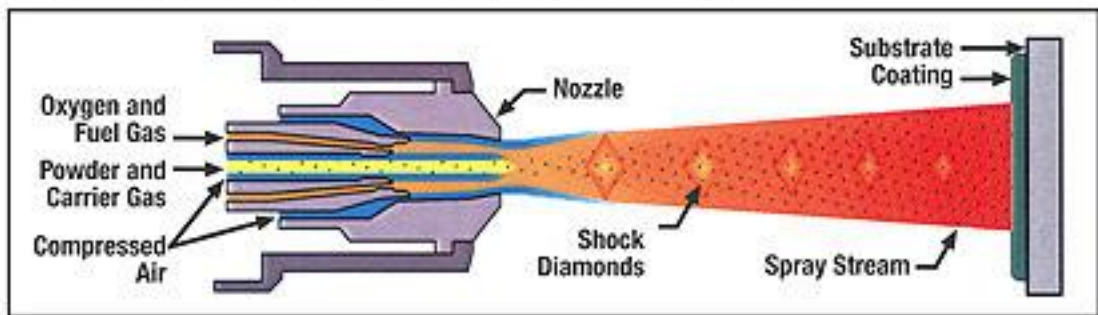


Fig 1.9: High velocity oxy-fuel process

1.4.5 EB-PVD:

In the EB-PVD process, the source material is heated with an electron beam, vapors are produced, and the evaporated atoms condense on the substrate. Crystal nuclei form on favored sites and grow laterally and in thickness to form individual columns that provide in-plane compliance. A TGO layer often forms on the bond coat in these TBC systems and increases the residual stress. Further, brittleness of the top coat increases with the sintering of the coating. Consequently, the adhesion of the bond coat to the top coat becomes weak at high temperatures. Therefore, the TGO layer is very detrimental for TBC performance.

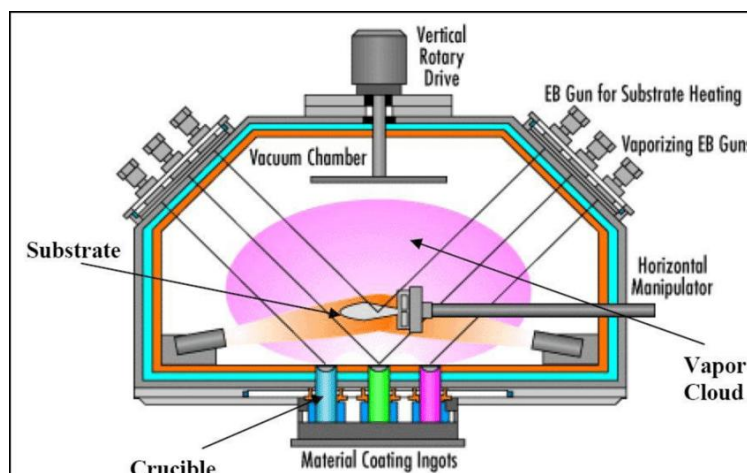


Fig 1.10: EB-PVD process

1.5. METHODS OF COATING'S CHARACTERIZATION

The coatings are characterized thoroughly by using many techniques. The simplest way of controlling the coating's quality is careful observation of its surface. The properties of coatings determine their behaviour during service. The best possible test is the one made under conditions simulating 'real-service' conditions. However, such a test is expensive and sprayed coatings are most frequently characterized by testing their physical or chemical properties.

1.5.1 Thermal Cyclic Test

Thermal Shock Resistance (TSR), also called thermal stress resistance, describes the capacity of a brittle material to resist rapid temperature variations without failure. The TSR depends on the conditions of heating (steady- state flow, transient heating, heating by convection or radiation, etc.) and may be expressed as the maximum allowable temperature difference in the body. In practice, TSR is tested for the system of a coating onto a substrate. Most of the tests simulate the conditions of the 20 service of the coatings. A typical thermal cycling furnace test rig is used to test coated turbine blades. Thermal Cyclic Fatigue Test was conducted by Furnace cycle test. In Furnace Cycle Test each thermal cycle consists of a 10-minute heat up to the steady-state temperature, a 45-minute soak at the steady-state temperature and a 5-minute forced air cool down. The chosen steady state temperature was 1150°C. The peel off determines the starting of the damage.



Fig 1.11: Thermal cyclic test

1.5.2 Hot Corrosion Test

Hot corrosion is a mechanism of "high temperature corrosion", which includes high temperature oxidation, sulfidation and carbonization. Hot Corrosion can be classified as hot corrosion by sulfates, Hot Corrosion by Vanadium, and Hot Corrosion by Lead. 21 Two types of sulfate-induced hot corrosion are generally distinguished. They are Type I and Type II. Type I takes place above the melting point of sodium sulfate and Type II occurs below the melting point of sodium sulfate but in the presence of small amounts of SO₃. In Type I, the protective oxide scale is dissolved by the molten salt. Sulfur is released from the salt and diffuses into the metal substrate forming discrete grey/blue colored aluminum or chromium sulfides so that, after the salt layer has been removed, the steel cannot rebuild a new protective oxide layer. Alkali sulfates are formed from sulfur trioxide and sodium-containing compounds. As the formation of vanadates is preferred, sulfates are formed only if the amount of alkali metals is higher than the corresponding amount of vanadium. The same kind of attack has been observed for potassium and magnesium sulfate. Vanadium is present in petroleum, especially from Canada, western United States, Venezuela and the Caribbean region, in the form of porphyrine complexes. These complexes get concentrated on the higher-boiling fractions, which are the base of heavy residual fuel oils. Residues of sodium, primarily from sodium chloride and spent oil treatment chemicals, are also present.

More than 100 ppm of sodium and vanadium will yield ash capable of causing fuel ash corrosion. Most fuels contain small traces of vanadium. The vanadium is oxidized to different vanadates. Furthermore, the presence of vanadium accelerates the diffusion of oxygen through the fused salt layer to the metal substrate; vanadates can be present in semiconducting or ionic form, where the semiconducting form has significantly higher corrosivity as the oxygen is transported via oxygen vacancies. Ionic form in contrast transports oxygen by diffusion of the vanadates, which is significantly slower. The semiconducting form is rich on vanadium pentoxide. At high temperatures or lower availability of oxygen, refractory oxides such as vanadium dioxide and vanadium trioxide were formed. These do not promote corrosion. However, vanadium pentoxide is formed at high temperatures. Various composition ratios of vanadates are formed with sodium oxide. Vanadates of composition approximating $\text{Na}_2\text{O} \cdot 6\text{V}_2\text{O}_5$ have the highest corrosion rates at the temperatures between 593°C and 816°C ; at lower temperatures the vanadate is in solid state, at higher temperatures vanadates with higher proportion of vanadium provide higher corrosion rates. 22 The solubility of the passivation layer oxides in the molten vanadates depends on the composition of the oxide layer. Iron (III) oxide is readily soluble in vanadates between $\text{Na}_2\text{O} \cdot 6\text{V}_2\text{O}_5$ and $6\text{Na}_2\text{O} \cdot \text{V}_2\text{O}_5$, at temperatures below 705°C which amounts equal to the mass of the vanadate. This composition range is common for ashes, which aggravates the problem. Chromium (III) oxide, nickel (II) oxide, and cobalt (II) oxide are less soluble in vanadates; they convert the vanadates to less corrosive ionic form and their vanadates are tightly adherent, refractory and acting as oxygen barriers. The corrosion rate by vanadates can be lowered by lowering the amount of excess air for combustion (thus forming preferentially the refractory oxides), refractory coatings of the exposed surfaces, or use of high-chromium alloys. Lead can form a low melting slag capable of fluxing protective oxide scales. Electrochemistry contributes an understanding of the mechanism that is basic to the corrosion of all metallic objects. Metallurgy provides knowledge of the characteristics of metals and their alloys as well as the methods of combining the various metals and working on them into the desired shapes.

a) Burner Rig Test In gas turbine,

Burner rig test has been developed to rank the coatings. The figure shows the ducted burner rig hot corrosion test. The essential features are the combustion chamber, means to ingest the salt and the specimen holder that is rotated and often periodically removed from the hot gas stream to induce thermal stress. The specimens are usually about 10 to 11 cm in length and about 1cm in diameter. In the burner rig test, the specimens are periodically removed from the test to measure weight changes or thickness and to be examined visually. Upon conclusion of the test the specimens are sectioned, metallographically prepared and examined using optical metallography and scanning electron microscope. It is also important to note that the testing in burner rigs is expensive. Furthermore, the construction and operation of burner rigs requires experienced designers and operators.

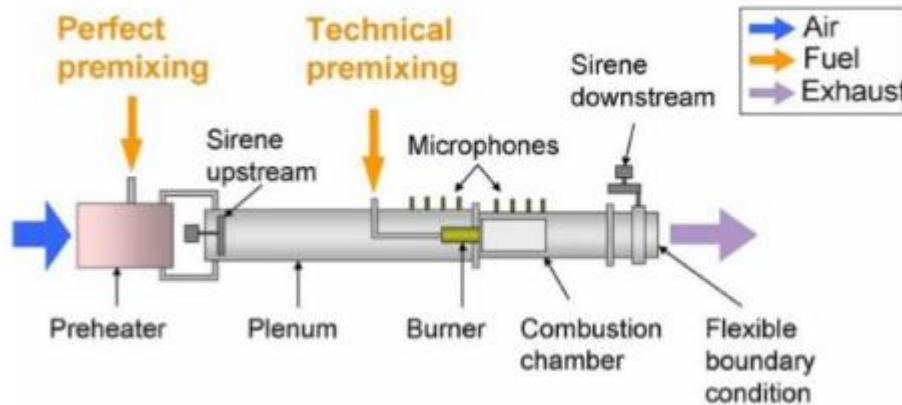


Fig 1.12: Burner Rig Test In gas turbine

b. Immersion test

In Immersion test, coupons are placed in deep melts. This type of test is usually used for deep melts. Immersion testing which was the first laboratory test method is not considered reliable for gas turbine environment

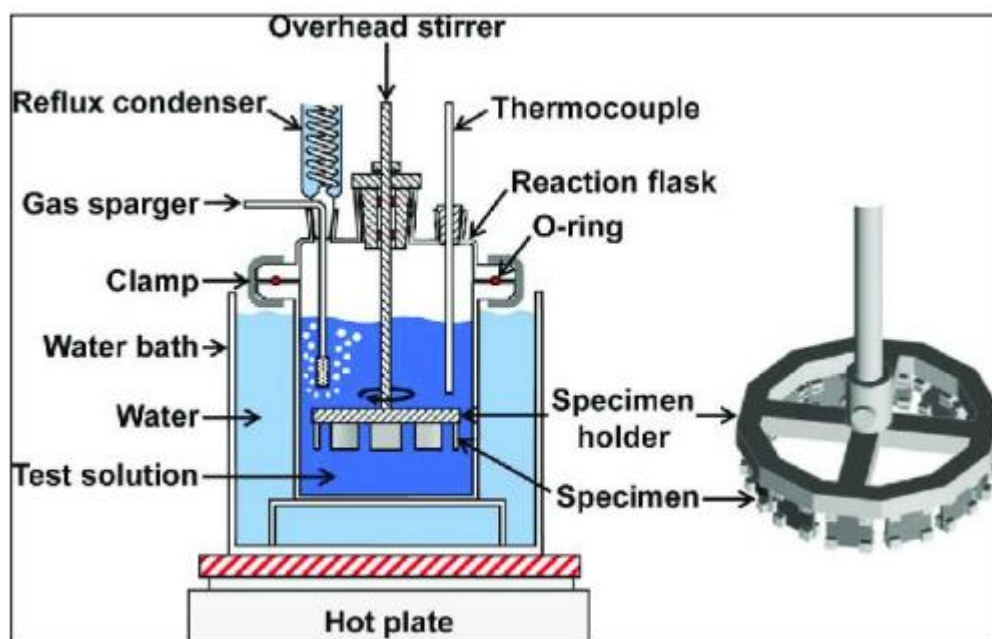


Fig 1.13: Immersion test apparatus

CHAPTER 2

LITERATURE SURVEY

- Dapeng Zhou et al., designed and deposited double-layered and triple layered APS YSZ/SPS GZO TBCs. Microstructures, thermal cycling performances and CMAS attack resistance of both TBCs systems were investigated. During thermal cycling test, both TBCs systems exhibited an extremely longer lifetime than conventional single-layered APS YSZ TBCs. This probably can be attributed to the high strain tolerance microstructures of TBCs and the better sintering resistance of GZO.
- Yasin Ozgurluk et al., exposed YSZ, Gd₂Zr₂O₇, YSZ/Gd₂Zr₂O₇ TBC systems to 45 wt.% Na₂SO₄ and 55 wt.% V₂O₅ molten salt mixtures at 1000 °C temperature. TBC samples were investigated and compared using scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) analysis and X-ray diffractometer (XRD). The hot corrosion failure mechanisms of YSZ, Gd₂Zr₂O₇ and YSZ/Gd₂Zr₂O₇ TBCs in the molten salts were evaluated. Hot corrosion molten salts severely damaged the column morphology of the EB-PVD method, reducing the thermal insulation properties and strain tolerance of TBCs.
- Satyapal Mahade et al., In this work, compared the erosion resistance at room temperature of standard 8YSZ single-layer TBC along with GZ/YSZ multi-layered TBCs that were deposited by suspension plasma spray process. Single-layer 8YSZ had a higher erosion resistance than GZ/YSZ multi-layered TBCs despite of its higher porosity content and slightly lower hardness among the as-sprayed TBCs.

A denser GZ top layer in the case of triple-layer TBC helped in slightly improving the erosion resistance compared to the GZ/YSZ double-layer TBC

- Mustafa Guven Gok et al., evaluated the production methods, design process, thermal and mechanical properties, failure mechanism, CMAS and hot corrosion resistance, and laser surface modification process of GZ-based TBCs. It was found that it is possible to further improve the thermal conductivity and thermal failure properties of the GZ-based TBCs by using different coating architectures such as multilayer and functionally graded designs. GZ-based TBCs have a significant advantage in terms of its CMAS and hot corrosion resistance.
- Satyapal Mahade et al., in their work found that although the failure modes under different thermal cyclic conditions differed for the investigated TBCs, durability was shown to be superior for 400GZ/100YSZ TBC. Further improvement in the durability of 400GZ/100YSZ TBC could be achieved by opting for a denser GZ microstructure (due to improved fracture toughness) as failure in BRT in this work was shown to be in the GZ layer close to the YSZ interface.
- Mustafa Guven Gok et al., in their study inferred that thermal shock life time of the GZ/CYSZ coatings having multilayered and functionally graded designs was longer than single layered GZ TBC. This situation was attributed to the decreasing thermal stress in the Mled and FGed GZ/CYSZ coatings due to the CYSZ having higher CTE than GZ. Results of the CMAS and hot corrosion tests showed that surfaces of YSZ and FG8 reacted differently with the CMAS + hot corrosion products.

- Bobzin.K et al studied the friction and wear behavior of NiCoCrAlY and mixture of NiCoCrAlY and yttria partially stabilized zirconia. The wear behavior was tested by pin on disc method. The abrasion rating of the coating produced with the NiCoCrAlY + 5 wt.% YPSZ powder was half of the rating of the commercial NiCoCrAlY coating. This showed that the ceramic mixed conventional coating has high wear resistance than the conventional NiCoCrAlY coating

CHAPTER 3

AIM AND SCOPE OF THE PRESENT INVESTIGATION

3.1. AIM

Evaluation of hot corrosion behavior on Yb₂O₃ doped Gd₂ Zr₂ O₇/ YSZ
Bilayer thermal barrier coatings on hastelloy x

3.2 OBJECTIVES OF STUDY

- In the growing field of aerospace engineering, where aircraft operate in aggressive environments characterized by high temperatures, pressure, stresses on individual components, and the presence of oxidizing and corroding atmosphere, the need for thermal barrier coatings is of utmost importance.
- Thermal barrier coatings (TBCs) applied to the surface of metallic parts in the hottest part of gas turbine engines enable advanced modern engines to operate at high temperatures well above 1200 °C enhancing the turbine engine efficiency.
- However, a major problem in thermal barrier coatings (TBC) applied to gas turbine components is the spallation of the coating under thermal cycling processes.
- Hence the major purpose of this study is to evaluate thermal behavior of as-deposited and aged GZ Thermal barrier coating (TBC) produced using Atmospheric Plasma Spray technique on Inconel 718 metal substrates

These include,

- To design and develop Yb₂O₃ doped GZ thermal barrier coating using air plasma spray coating.
- To investigate the hot corrosion behaviour of thermal barrier coating by hot corrosion test. (55 wt.% Na₂SO₄ and V₂O₅)
- To perform isothermal oxidation test and thermal cyclic fatigue test to determine the life of the coating

CHAPTER 4

MATERIALS AND METHODS

4.1 EXPERIMENTAL PROCEDURE – SCHEME OF RESEARCH

The objective of this investigation is to the major purpose of this study is to evaluate the tribological and thermal behavior of as-deposited and aged GZ Thermal barrier coating (TBC) produced using Atmospheric Plasma Spray technique on Inconel 718 metal substrates.



Figure 4.1: Methodology for developing thermal barrier coating

4.2 SELECTION OF BASE MATERIAL

Super alloys are metallic materials for service at high temperatures, particularly in the hot zones (turbine blades) of gas turbines. Such materials allow the turbine to operate more efficiently by withstanding higher temperatures. These high-pressure turbine blades are made up of Nickel and Chromium. Since Inconel contains Nickel and Chromium and also is commercially available, the base metal is taken as Inconel 718.

Table 4.1 Chemical Composition (wt%) of the base metal

Elements	C	Mn	Si	Ti	Al	Co	Mb	Cb	Fe	Cr	Ni
Percentage	0.08	0.35	0.35	0.6	0.8	1.0	3.0	5.0	17.0	19.0	52.82

Figure 4.2:Chemical composition of base metal

4.3 SPECIMEN PREPARATION

4.3.1 Surface Cleaning

Degreasing of new metal substrates was done by using organic solvent acetone. The process was carried out in a clean room and the remaining liquid was carefully recycled. Every care was taken to protect the operator from hazardous exposure.

4.3.2 Substrate Shaping

The substrates need to be shaped in order to meet the dimensional specifications. A little material has to be removed in the places where the coating is to be deposited. Shaping is also necessary to avoid sharp angles or sharp ends where it is difficult to spray a coating or, on the other hand, where adherence to the substrate is poor. The adherence becomes lower because of the high concentration of thermal stresses inside the coatings sprayed in such places. Substrate shaping was done by wire cutting method.

4.3.3 Surface Activation

Surface roughness is the parameter which is closely related to grit blasting and, on the other hand, strongly influences the adhesion of the coating to the substrate. An increase in the roughness results in an increase in the adhesion.

Nozzle-GH	PRESSURE-PSI	FLOW RATE-SEFH
Argon	100-200	80-90
Hydrogen	50	15-18
Current	500 amps	65-70 volts
Powder feed	40-45 grains/min	
Spray distance	2-3 inches	

Figure 4.3

4.4 PRE-TREATMENT PROCESS

A bond coating of NiCrAlY was used in the thermal barrier coatings. The bond coating was sprayed by plasma spray process. An improvement in oxide coating adhesion can be achieved by the application of bond coatings. The first layer of the coating is called the bond coat. It enables the coating to bond over the substrate and therefore plays an integral role in forming a thermal barrier.

4.5 PLASMA SPRAY COATING

Among the different coating systems, the plasma spray process is commonly used to protect the hardware operating in high temperature environments, such as combustor liners and gas turbine blades, from excessive high heat fluxes and temperatures.

Plasma spraying uses metal in powder form. The ceramic coating system is shown in figure. The coating materials in the form of a powder is passed into a plasma jet in which powder particles are melted and accelerated towards the surface to be coated.

The powder particles, approximately 50 micrometers in diameter, are accelerated and melted in the flame on their high speed 200 m/sec path to the substrate, where they impact and undergo rapid solidification.

4.6 PLASMA SPRAY COMPOSITION

The optimum coating composition has to be identified in order to withstand the high temperature and resist corrosion. Hence the three various compositions were taken into consideration.

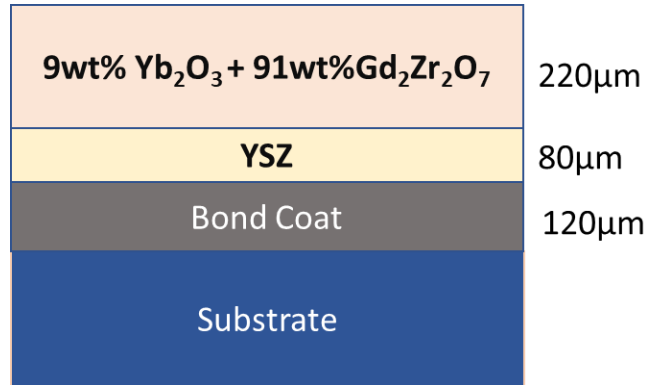


Figure 4.4: Bi-Layer 9YbGZ TBC

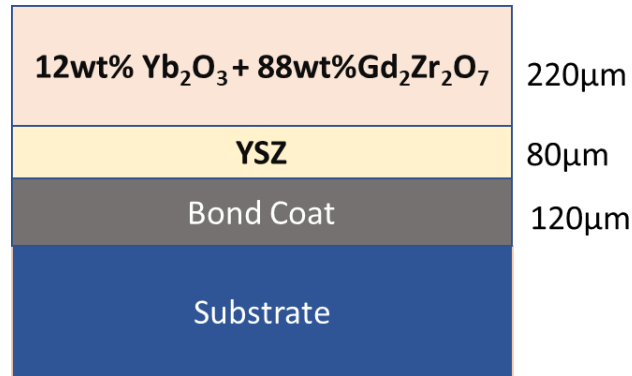


Figure 4.5: Bi-Layer 12YbGZ TBC

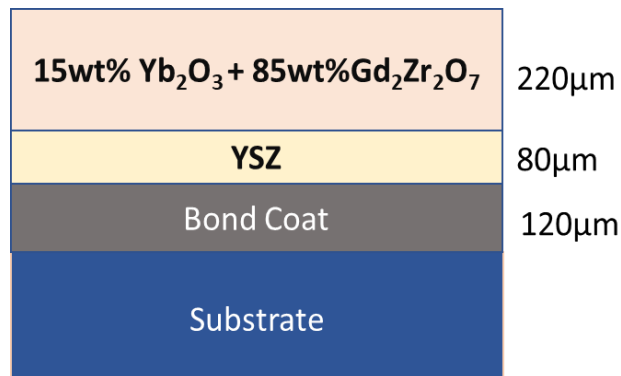


Figure 4.6: Bi-Layer 15YbGZ TBC

The samples were coated using Atmospheric plasma spray process as shown in the above diagrams.

4.7 TESTING OF COATINGS

After the completion of coatings over the samples, they were subjected to various tests such as,

- 1) Isothermal oxidation test
- 2) Thermal cyclic fatigue (TCF)
- 3) Hot corrosion

4.7.1 Isothermal oxidation test:

Isothermal oxidation test is carried out using a muffle furnace to study the phase changes and life of the coating after prolonged heating. The samples are placed in the furnace and heated to 1150 degrees and this temperature is maintained throughout the experiment. The coatings are observed every 50 hours by opening the furnace and inspecting visually and closing the door without any loss in furnace temperature. The experiment was continued until the 20 percent failure of the coating was observed.

4.7.2 Thermal Cyclic Fatigue Test

Thermal cyclic fatigue test was performed on a muffle furnace as shown in figure. According to the OEM specification, the minimum required number of thermal cycles to pass the thermal cycling fatigue test is 250 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 carried out confirming to ASTM C 385-58 standards. Initial weights of each coated samples were noted. Coated samples were kept inside the furnace.



Figure 4.7 :Thermal Cyclic Fatigue Test

The coated samples were heated to steady state temperature of 1150°C within 10 minutes and maintained for 45 minutes. The samples were cooled to room temperature within 5 minutes. The cooling was done by air. The weight of the coated samples subjected to thermal cyclic test was measured after each cycle. The cycle was repeated for 250 times. Final weight losses were measured after 250 cycles.

4.7.3 Hot Corrosion Test

Hot corrosion test was performed by controlled laboratory test. Controlled laboratory test (Thin salt film test) has been performed using conditions typical of gas turbine engines. Furnace has been used with controlled composition of flowing gases where the salt is deposited on the specimen. In this type of testing, the conditions were well defined and precisely controlled. The laboratory test used for high temperature hot corrosion consisted of cyclic oxidation using application of Na₂SO₄. The specimens were small coupons. The specimens were cycled from the test temperature to room temperature every hour. The gas was replaced by air.

The specimens were weighed and visually examined at selected times. Based upon weight changes and visual examination, the specimens were removed from the test when the degradation progressed to failure limits. In this research work, controlled laboratory test was followed. The hot corrosion test was carried out confirming to ASTM G41.



Figure 4.8: Hot Corrosion Test

In hot corrosion test, Na_2SO_4 and V_2O_5 were selected as corrosive salt. Accurate dimensions were taken by digital vernier to calculate the area of ten specimens of each coating composition.

The Na_2SO_4 and V_2O_5 Molten salt hot corrosion test was performed on all the coatings. 55wt % Na_2SO_4 and 45 wt% of V_2O_5 were mixed together with small amount of distilled water to form a salt paste.

The corrosive salt paste was spread over the surface of the coating in a concentration of 4 mg/cm² on the preheated samples (250°C). The initial weight was measured. After the salt application, the specimens were put in an electric furnace with air atmosphere as shown in figure.

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 COATING

All the substrates were deposited with NiCrAlY bond coat and YSZ and then deposited with 9YbGZ , 12YbGZ and 15YbGZ to produce the bilayer Yb₂O₃ doped gadolinium zirconate doped TBC. The substrate thickness were measured before coating and labeled and tabulated. And the thickness after deposition of coating was measured tabulated. The difference gives is the coating thickness. All the substrates were coating to average thickness of 250 to 300 microns approximately. The coated samples then are subjected to thermal analysis.

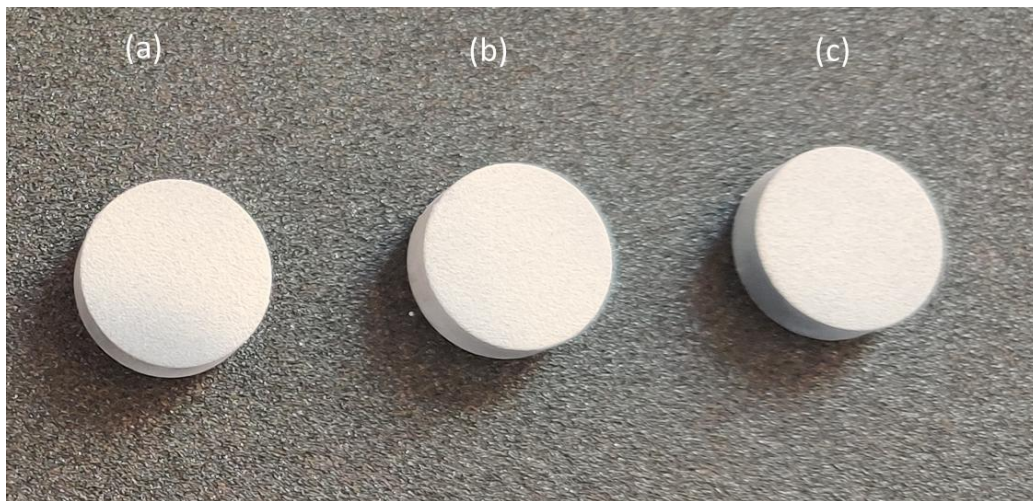


Figure 5.1

5.2 ISOTHERMAL OXIDATION TEST

The coated substrates were subjected to thermal oxidation in a furnace 1150 degrees Celsius for 500 hours. All the coatings did not show any sign of degradation until 400 hours.

9YbGZ bilayer coating started developing a swelling along one side of the substrate after 450 hours and after 480 hours more than 20 % spallation that is a major crack was observed in the coating. And was removed from the experiment.

The other 12YbGZ bilayer coating did not show any sign of degradation until 500 hours but suddenly degraded and more than 20 % failure of the coating was observed after 520 hours of isothermal oxidation test. Coating failure immediately without showing any signs of degradation prematurely. Where as in case of 9YbGZ bilayer coating failure mechanism was creep failure as the integrity of the coating was reduced over course of 80 hours.

The 15YbGZ bilayer coating did not show any sign of failure even after the 600 hours of isothermal oxidation test.

5.3 THERMAL CYCLIC FATIGUE TEST

The coated samples were placed in a muffle furnace preheated to 1150 degrees Celsius and the coatings are heated for 55 minutes and immediately removed from the furnace and cooled using pressurized air and brought to room temperature within 5 minutes this cycle was repeated until 20 percent failure of the coating was observed.

9YbGZ bilayer coating was first coating show signs of failure the earliest the coating started developing crack along the circumference of the disc after 250 hours and after 260 cycles more than 30 % failure of the coating was observed. 12YbGZ bilayer coating and 15YbGZ bilayer coating did not show any sign of failure until 300 cycles.

After 320 cycles 12YbGZ bilayer coating developed a swelling along the edge and failed after 225 cycles. 15 YbGZ bilayer coating developed a crack along the after 350 hours and 20 % spallation was observed after 370 hours and the experiment was concluded.

5.4 HOT CORROSION TEST

The Na_2SO_4 and V_2O_5 Molten salt hot corrosion test was performed on all the coatings. 55wt % Na_2SO_4 and 45 wt% of V_2O_5 were mixed together with small amount of distilled water to form a salt paste.

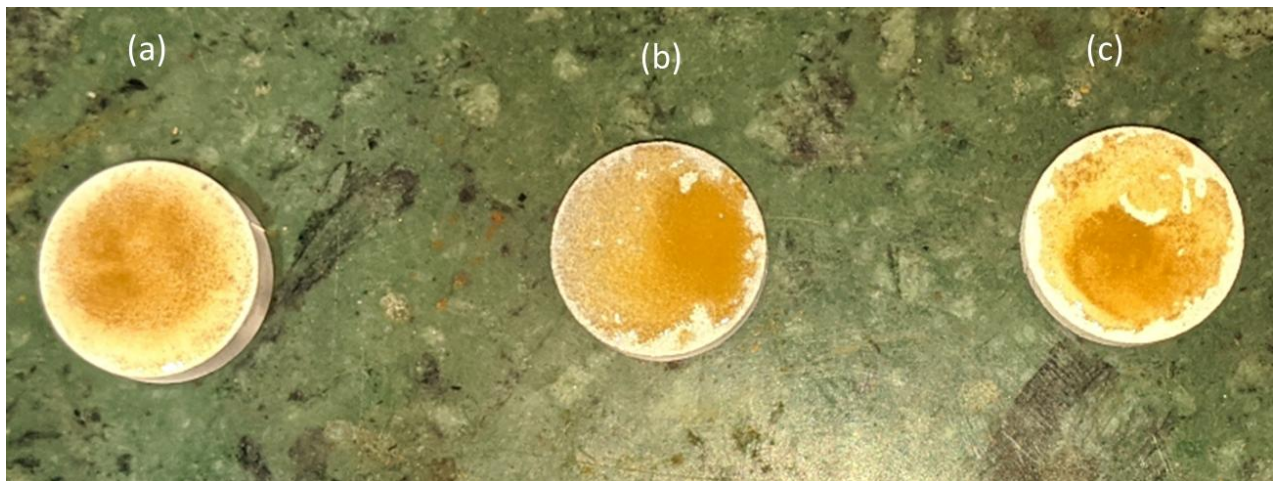


Figure 5.2: Coating after applying salt paste (a) 9YbGZ bilayer TBC (b) 12YbGZ bilayer TBC (c) 15YbGZ bilayer TBC

This was applied on the surface of the coating. And the samples were placed in the furnace and heated to 1273 K and observed for failure every 5 hours.

All the coating withstood the experiment for 30 hours without any sign failure and all the coatings failed immediately after 50 hours. All the coatings have excellent and very similar hot corrosion behavior.

5.5 CONCLUSION

Yb₂O₃ was selected a dopant to improve the thermal behaviour of gadolinium zirconate thermal barrier coating. The GZ was doped with 9%, 12% and 15% Yb₂O₃ using ball mill and the doped powder. the hastelloy X substrate of 20 mm dia substrates were deposited with NiCrAlY Bond coated to a thickness of 100 to 120 microns and then YSZ was deposited onto the bond coat to a thickness of 80 microns. and then Yb₂O₃ doped powders were deposited to a thickness of 180 to 220 microns to a overall thickness of 300 to 350 microns. The TBCs were subjected to isothermal oxidation test , thermal cyclic test and hot corrosion test and the conclusions are as follows.

- Isothermal oxidation test revealed that all the Yb₂O₃ doped GZ bilayer coatings have excellent oxidation stability compared to reference GZ TBC. and especially 15YbGZ bilayer TBC outperforms 9YbGZ and 12YbGZ.
- Similarly in thermal cyclic fatigue test increase in the dopant concentration improves the cyclic life of the coating. 9YbGZ bilayer TBC failed earlier than 12YbGZ and 15YbGZ was the last to show 20% degradation.
- All the bilayer coating have excellent hot corrosion resistance compared to undoped GZ. All the coatings failed at the last hour of the testing but the 15 YbGZ showed minimum degradation compared to others.

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