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PROCEEDINGS OF JOHOR INTERNATIONAL INNOVATION INVENTION COMPETITION AND SYMPOSIUM 2024 (JIICaS 2024)



*“Flourish and Nurturing Sustainable
Innovation for a Prosperous Nation”*

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Preface

In the name of Allah, the Almighty who gives us the enlightenment, the truth, the knowledge and with regards to Prophet Muhammad (peace be upon him) for guiding us to the straight path. We thank to Allah for giving us guidance and strength to write this e-book.

This e-book compiles the extended abstracts that submitted to Johor International Innovation Invention Competition and Symposium 2024 (JIIICaS2024), where JIIICaS2024 is a virtual platform for all creative minds to share and present their invention and innovation. Each abstract gives a brief background on the innovation or project.

We hope that this e-book will help the readers to get to know the innovation done by the students and get some ideas to develop future innovation products.



Foreword Rector



Assalamualaikum warahmatullahi Wabarakatuh,
Salam Sejahtera, Salam Malaysia MADANI and
Salam UiTM Dihatiku.

In the name of Allah, the Most Gracious, the Most
Merciful.

It is a great honor to welcome you to the Johor
International Innovation, Invention, Competition, and
Symposium 2024 (JIIICaS 2024). This event

connects various disciplines, focusing on education and engaging educators,
students, researchers, and innovators from all walks of life.

Innovation is not just about ideas; it demands perseverance, creativity, and
determination to turn those ideas into reality. The remarkable projects
showcased today highlight the dedication and spirit of all participants.
Initiatives like this not only explore new technologies but also cultivate skills
and leadership among our youth. At Universiti Teknologi MARA (UiTM) Johor
Branch, we are fully committed to fostering a dynamic culture of innovation,
promoting the commercialization of new products, and encouraging
meaningful collaborations with industry and society.

As we celebrate this event, I would like to extend my heartfelt gratitude to all
sponsors, judges, the College of Computing, Informatics and Mathematics,
UiTM Pasir Gudang Campus as the event organizer, as well as to the
researchers and participants for their hard work in making this event a
success. Let us continue striving for innovation and excellence. May the
ideas presented today inspire us and lay the groundwork for future
achievements.

Thank you.

Associate Professor Dr. Saunah Zainon
Rector
Universiti Teknologi MARA (UiTM)
Johor Branch

(A-ST131) ZIRCONATE-BASED DOUBLE CERAMIC LAYER SYSTEM OF THERMAL BARRIER COATINGS (TBC)

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ABSTRACT

Ceramic coatings used in high-temperature applications, focusing on their behaviour under thermal stress and their ability to resist oxidation. The aim of this project is to evaluate on how these coatings can prevent cracks caused by oxidation at the thermally grown oxide (TGO) layer. Two types of samples which single ceramic layer and double ceramic layer thermal barrier coating (TBC) were prepared using Atmospheric Plasma Spray (APS). The high temperature oxidation was conducted on ceramic-coated samples exposed to 600°C in electric furnace and subsequent cooling. The outcomes of this study will contribute to the development of ceramic coatings with enhanced resistance to oxidation, particularly in high-temperature environments. The findings provide insights into the factors influencing the formation and growth of TGO layer and contribute to the prevention of cracks caused by oxidation. The study compared the oxidation resistance of single-layer YSZ (Yttria-Stabilized Zirconia) and double-layer LZ/YSZ (Lanthanum Zirconate/Yttria-Stabilized Zirconia) thermal barrier coatings (TBCs) under high-temperature conditions (600°C). The findings revealed that the double-layer LZ/YSZ coating demonstrated better resistance to oxidation, as indicated by lower weight changes and thinner thermally grown oxide (TGO) layers, compared to the single-layer YSZ coating. This suggests that the LZ/YSZ coating offers improved protection against oxidation and better performance in extreme temperature environments. This research holds practical implications for industries relying on high-temperature applications. It establishes a foundation for further advancements in ceramic coatings, with the objective of improving material performance and extending the lifespan of components operating in extreme conditions.

Keywords: Ceramic coatings, thermal stress, oxidation resistance, thermally grown oxide (TGO) layer, high-temperature applications

Introduction

In recent years, there has been a great demand for the development of materials used for ceramic coatings' pivotal role in high-temperature applications such as gas turbines, where their microstructure significantly influences properties and performance. The study emphasizes the use of ceramic coatings, LZ and YSZ, on Inconel 625 to enhance its durability and resistance to oxidation. Recognizing the challenges faced by turbine blades in harsh conditions, the research aims to characterize the microstructure of Single Ceramic Layer (YSZ) and Double Ceramic Layer (LZ/YSZ) coatings, shedding light on their structural integrity and resistance to high-temperature degradation. The problem statement underscores concern about TBC degradation, prompting the need for innovative coating materials and designs to improve gas turbine engine durability and safety.

Objective

The research objectives encompass microstructural characterization before and after testing, evaluation of thermally grown oxide (TGO) growth, and assessment of high-temperature oxidation behaviour. The expected outcomes include insights into oxidation prevention, the effectiveness of double-layer coatings, and the protective role of TGO, contributing to the optimization of ceramic coatings for high-temperature applications

Methodology

Material

The Inconel 625 was cut into pieces measuring 15 x 15 x 6 mm. The double ceramic layer (DCL) LZ/YSZ coating system was prepared using APS with specific spraying parameters outlined in Table 2. Specimens were sandblasted with 24–50 mesh alumina grit to increase surface area and improve coating adhesion, resulting in a surface roughness of 6–8 μm , measured with a Mitutoyo surface roughness tester. The roughened surfaces were then cleaned with acetone in an ultrasonic bath for 15 minutes, washed, and preheated at 70–100 $^{\circ}\text{C}$ in an oven. A bond coat of NiCoCrAlYTa powder (particle size 5–37 μm) was applied using a high-velocity oxy-fuel (HVOF) system, achieving a thickness of 150 μm . For the topcoats, Metco 204 NS-G YSZ powder (ZrO_2 -8 wt% Y_2O_3) was applied using atmospheric plasma spray (APS) deposition equipment as the intermediate layer, resulting in a thickness of 200–250 μm . Finally, commercial $\text{La}_2\text{Zr}_2\text{O}_7$ powder was used as the topcoat, achieving a thickness of 80–100 μm .

Table 2 Air plasma spray parameters.

Parameters	YSZ	LZ
Current (A)	600	600
Voltage (V)	70	70
Primary gas, Ar (l/min)	38	35
Secondary gas, H ₂ (l/min)	3	8
Powder feed rate (g/min)	35	34
Spray distance (cm)	12	10

Experimental Setup

Designing a furnace setup for high-temperature oxidation tests involves several key components and specifications:

- i. **Furnace Dimensions:** Must accommodate the test specimens and ensure uniform heating and cooling based on test requirements.
- ii. **Insulation Materials:** High-temperature insulation materials like ceramic fibre or refractory bricks minimize heat loss and maintain a stable environment.
- iii. **Heating Elements:** Typically, resistance heating elements such as Kanthal or Nichrome are used to achieve and sustain the target temperature of 600 $^{\circ}\text{C}$.
- iv. **Temperature Control System:** Employs sensors and feedback mechanisms for precise temperature control throughout the test, including heating to 600 $^{\circ}\text{C}$ for 5 hours and cooling for 24 hours.

These components ensure a controlled environment for accurate assessment of materials performance under cyclic high-temperature stress.

Sample Preparation

In the high-temperature oxidation test, rectangular specimens of Inconel 625 are used, each measuring 15mm x 15mm x 6mm. One specimen is coated with LZ and weighs 10.7063g, while another is coated with YSZ/LZ and weighs 11.3637g. The dimensions (length, width, and height) and weight of the specimens at Figure 0.1 are critical for assessing their response to high-temperature oxidation. The surface area, determined by length and width, influences the interaction with the high-temperature environment. The volume, derived from the dimensions, helps understand the material's oxidation behaviour and volume changes. The weight is used to calculate heat capacity, which provides insights into the energy required for temperature elevation and thermal energy storage. These parameters collectively help evaluate the oxidation rate, extent of oxidation, and performance variations based on different coatings.



Figure 0.1 Specimen size: 15mm x 15mm x 6mm.

High Temperature Oxidation test

The pre-oxidation process involves placing specimens in a furnace for about 12 hours at an elevated temperature to form a consistent oxide layer. Before testing, the specimens are meticulously prepared through cleaning, deburring, coating, or adjusting surface roughness to ensure accuracy. The furnace is then configured and calibrated for precise temperature control, and specimens are placed inside to ensure uniform heating and cooling. The heating cycle begins with a gradual increase in temperature, typically at a rate of 8°C per minute, from ambient to the desired maximum. This is followed by a cooling cycle, where the temperature is decreased back to ambient conditions at the same rate. To replicate real-world conditions, multiple heating and cooling cycles are performed. During the process, researchers closely monitor and record parameters such as temperature, time, and specimen responses. Specific heating and cooling cycles might involve, for instance, heating to 600°C for 5 hours followed by a 24-hour cooling period. Additionally, dwell periods or hold times may be introduced at temperatures to allow for stabilization or observation, depending on the testing requirements and the properties of the materials being tested.

Result

Microstructure

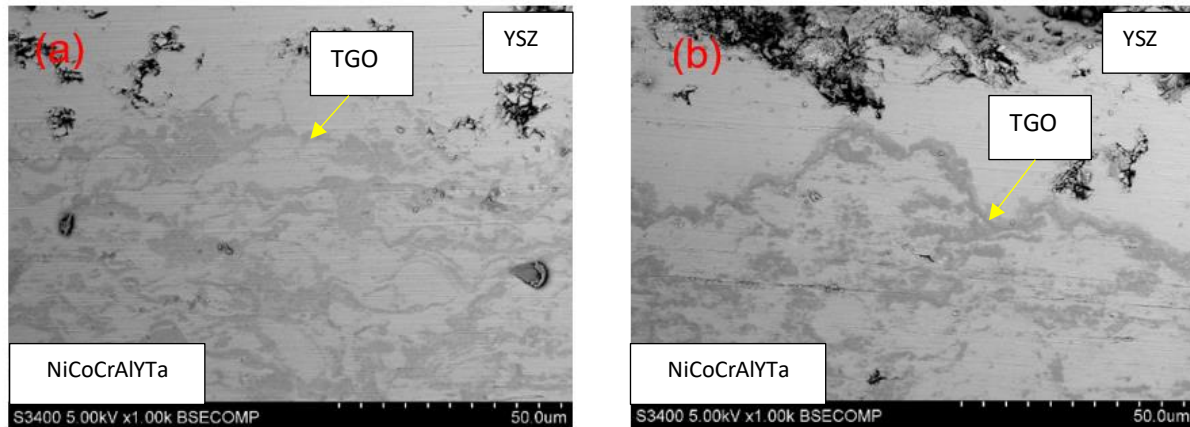


Figure 0.1 SEM Microstructure of sample (a) Lanthanum Zirconia/ Yittria Stabilized Zirconate (b) Yttria Stabilized Zirconate.

Figure 0.1 highlights the oxidation behaviour of dual-layer (DCL) and single-layer (SLC) coatings after a 90-hour high-temperature oxidation test. The DCL coating exhibits superior oxidation resistance, as evidenced by the absence of a black area representing the Thermally Grown Oxide (TGO) layer. Instead, the presence of a grey area within the DCL coating indicates mixed oxides, including chromia, spinel, and nickel oxides (CSN), suggesting a complex but effective defence against oxidation. In contrast, the single-layer YSZ coating shows a clear TGO line after 90 hours, indicating less robust oxidation resistance. The cross-sectional morphology of the YSZ coating reveals a continuous Al_2O_3 scale (black area) and lacks the mixed oxides seen in the grey area of the DCL coating. This distinction underscores the unique oxidation characteristics of the YSZ coating during high-temperature oxidation testing and emphasizes the influential role of layer thickness ratios in determining the overall oxidation resistance of the coatings.

10 Hours

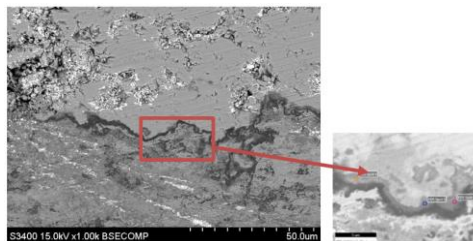


Figure 0.2 Close up TGO layer for YSZ

Table 3 Data of Elements in TGO layer YSZ

Coloured	Element	%Atomic
Dark Area	Al ₂	20.42
	O ₃	42.68
Gray Area	Cr	17.92
	Co	8.36
	Ni	10.62

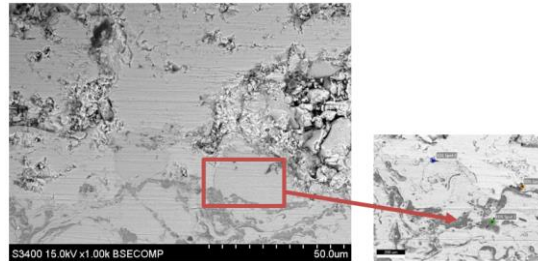


Figure 0.3 Close up TGO layer for LZ/YSZ

Table 4 Data of elements in TGO layer LZ/YSZ

Coloured	Element	%Atomic
Dark Area	Al ₂	30.5
	O ₃	28.98
Gray Area	Cr	14.55
	Co	14.66
	Ni	11.31

90 Hours

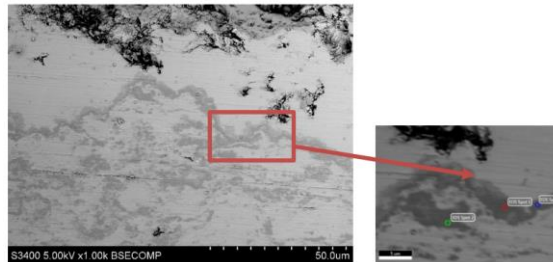


Figure 0.4 Close up TGO layer YSZ.

Table 5 Data of Elements in TGO layer of YSZ

Coloured	Element	%Atomic
Dark Area	Al ₂	18.77
	O ₃	31.98
Gray Area	Cr	19.01
	Co	14.3
	Ni	15.94

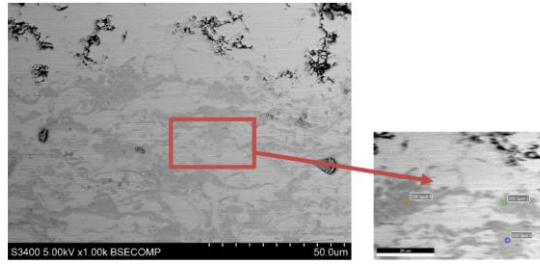


Figure 0.5 Close up TGO layer for LZ/YSZ

Table 6 Data of elements in TGO layer of LZ/YSZ

Coloured	Element	%Atomic
Dark Area	Al ₂	20.55
	O ₃	30.28
Gray Area	Cr	16.55
	Co	14.84
	Ni	17.78

The elemental composition analysis of the coated sample over 10 and 90 hours reveals significant changes in both dark and grey areas, reflecting material transformation over time. In the dark area, representing the primary coating, the percentage of Al₂O₃ decreased from 20.42% to 18.77%, and O₃ decreased from 42.68% to 31.98%, indicating ongoing oxidation processes. In the grey area, which includes mixed oxides like Cr, Co, and Ni, notable increases were observed: Cr from 17.92% to 19.01%, Co from 8.36% to 14.3%, and Ni from 10.62% to 15.94%. These changes suggest dynamic evolution due to oxidation and transformation during extended heat treatment. The increased percentages of Cr, Co, and Ni highlight their importance in the evolving composition of the grey area. Overall, this analysis provides valuable insights into material changes during high-temperature oxidation testing, enhancing understanding of the coating's behaviour over time.

Weight gain



Figure 0.6 Graph represent Weight Gain and Lost of Sample

Figure illustrates the weight changes over a 90-hour period for LZ and YSZ samples. The y-axis depicts weight in a narrow range from -0.003 to 0.003, while the x-axis represents time in hours (10 to 90). Peaks and troughs in the graph indicate variations in weight gain and loss, potentially associated with oxidation processes. Peaks suggest points of interest, likely indicating thickness increase during specific cycles, while weight loss at certain hours may signify processes resulting in reduced weight. The data implies that these specific hours are crucial for assessing sample performance. LZ, with a double coating layer (LZ/YSZ), outperforms YSZ with a single

layer, indicating more stable weight changes over the 90-hour period during high-temperature oxidation testing.

TGO Thickness

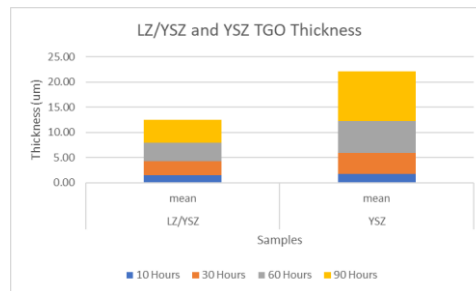


Figure 0.7 Comparative Analysis of LZ/YSZ and YSZ Samples by Layer Thickness in Micrometres (μm) with Mean Values

The provided stacked bar chart compares the thickness of thermally grown oxide (TGO) layers in LZ/YSZ and YSZ samples over four-time intervals: 10, 30, 60, and 90 hours. The mean thickness in micrometres (μm) shows an increase over time for both samples, consistent with TGO growth due to high-temperature oxidation. The YSZ sample consistently exhibits a significantly greater mean thickness across all time intervals compared to the LZ/YSZ sample. After 90 hours, the YSZ sample has the highest increase in TGO thickness, indicating a faster oxide growth rate or a different oxidation mechanism. This information is crucial for high-temperature oxidation testing, as TGO growth rates affect the performance and lifespan of protective coatings in turbines, engines, and other thermal barrier systems.

Conclusion

This study aimed to investigate key aspects related to the microstructure, Thermally Grown Oxide (TGO) growth, and the influence of heat treatment on different ceramic coating configurations, specifically comparing single-layer YSZ (Yttria-Stabilized Zirconia) coatings with double-layer LZ/YSZ (Lanthanum Zirconate/Yttria-Stabilized Zirconia) coatings. The results obtained shed light on the distinctive characteristics and performance of these coatings under high-temperature oxidation testing conditions.

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