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MICROSTRUCTURAL CHARACTERIZATION OF CERAMIC COATINGS FOR HIGH-TEMPERATURE APPLICATION

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ABSTRACT

Ceramic coatings are used in high-temperature applications, focusing on their behaviour under thermal stress and their ability to resist oxidation. The aim is to understand how these coatings can prevent cracks caused by oxidation on the thermally grown oxide (TGO) layer. Tests were conducted on ceramic-coated samples exposed to cycles of high temperatures (600°C) and subsequent cooling. The duration of pre-oxidation was varied, and two types of samples single ceramic layer and double ceramic layer TBC were used. The research aims to identify the time it takes for oxidation to occur after applying ceramic coatings and evaluate whether double-layer coatings offer superior protection against oxidation compared to single-layer coatings. The outcomes ofthis study will contribute to the development of ceramic coatings with enhancedresistance to oxidation, particularly in high-temperature environments. The findings provide insights into the factors influencing TGO layer thickness and contribute to the prevention of cracks caused by oxidation. This research holds practical implications for industries relying on high-temperature applications. It establishes a foundation for further advancements in ceramic coatings, intending to improve material performance and extend the lifespan of components operating in extreme conditions.

1.0 Introduction

In this introduction, the focus is on ceramic coatings' pivotal role in high-temperature applications, 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 [1][2].

The research objectives encompass microstructural characterization, evaluation of thermally grown oxide (TGO) growth, and assessment of high-temperature oxidation behavior[3]. The expected outcomes include insights into oxidation prevention, the effectiveness of double-layer coatings, and the protective role of TGO[4], contributing to the optimization of ceramic coatings for high-temperature applications [5].

2.0 Research Methodology

2.1 Experimental Setup

In designing a furnace setup for the high-temperature oxidation test, several crucial components and specifications must be considered[6]. Firstly, the dimensions of the furnace need to accommodate the test specimen, ensuring uniform heating and cooling. These dimensions are selected based on the specific requirements of the test setting. Secondly, high-temperature insulation materials, such as ceramic fibre or refractory bricks, are essential to minimize heat loss and maintain a stable environment within the furnace. This insulation ensures efficient heating during the test. Thirdly, the furnace is equipped with suitable heating elements, typically resistance heating elements like Kanthal or Nichrome, capable of achieving and sustaining the target temperature of 600°C.

Additionally, a robust temperature control system, employing sensors and feedback mechanisms, ensures precise temperature control throughout the test, which involves heating to 600°C for 5 hours and subsequent cooling for 24 hours. The heating and cooling rates are determined by the time required to reach and maintain the desired temperature during each phase, providing a controlled environment for accurate assessment of materials' performance under cyclic high-temperature stress.

Figure 1: Carbolite AAF 11/18 Furnace

2.2 Sample Preparation

The high-temperature oxidation test uses rectangular specimens measuring 15mm x 15mm x 6mm, where the length and width are 15mm, and the height is 6mm. The Inconel 625 specimen coated with LZ weighs 10.7063g, while the Inconel 625 specimen coated with YSZ/LZ weighs 11.3637g. These dimensions and weights are crucial for evaluating the specimens' response to high-temperature oxidation. The length and width determine the surface area, influencing the interaction with the elevated temperature environment.

The volume, derived from dimensions, helps understand how the material undergoes oxidation and associated volume changes. The mass, indicated by weight, is essential for calculating heat capacity, providing insights into energy requirements for temperature elevation and thermal energy storage. Together, these parameters contribute to understanding the specimens' behaviour, oxidation rate, extent of oxidation, and potential variations in performance based on different coatings during high-temperature oxidation testing.

Figure 2: Specimen size: 15mm x 15mm x 6mm.

2.3 Test Procedure

2.3.1 Pre-oxidation Process

In this initial step of the high-temperature oxidation test, specimens are placed within the furnace for a predetermined duration, typically 12 hours, at an elevated temperature. The purpose of this pre-oxidation process is to establish a consistent oxide layer on the specimen surfaces. This oxide layer plays a crucial role in influencing the subsequent behaviour of the specimens during the main high-temperature oxidation test.

2.3.2 Specimen Preparation

Before entering the test phase, meticulous attention is given to the preparation of specimens. This involves ensuring the specimens are thoroughly cleaned, free from contaminants, and undergo any necessary surface treatment steps. Common preparatory actions include deburring, coating application, or adjustments to surface roughness to facilitate accurate and reliable testing conditions.

2.3.3 Furnace Setup

The furnace setup is a critical component of the testing procedure. It involves configuring the furnace according to specifications, considering dimensions, insulation, and heating elements. Additionally, the temperature control system is calibrated to ensure precise and reliable operation throughout the test. This step ensures a controlled environment for accurate evaluation[7].

2.3.4 Specimen Placement

Once the furnace is ready, the prepared specimens are carefully positioned within it. Proper spacing is ensured to facilitate uniform heating and cooling. The correct placement of specimens within the furnace is vital for obtaining reliable and representative test results, as it influences the heat distribution and overall performance.

2.3.5 Heating Cycle

The actual test begins with the initiation of the heating cycle. The temperature is gradually increased from ambient conditions to the desired maximum temperature. The heating rate is adjusted according to the specific test requirements, for instance, setting it at 8 degrees Celsius per minute. This phase aims to simulate the conditions of elevated temperature exposure.

2.3.6 Cooling Cycle

Following the heating cycle, the cooling cycle is implemented. The temperature is gradually reduced from the maximum level back to ambient conditions. The cooling rate is adjusted as per the test requirements, such as 8°C per minute. This phase simulates realistic thermal stress conditions, allowing researchers to observe the specimens' response to temperature variations.

2.3.7 Repeat Cycles

To replicate real-world scenarios and enhance the reliability of the test, multiple heating and cooling cycles are conducted. The number of cycles is determined based on the material's anticipated operating conditions and the specific objectives of the test. This step ensures a comprehensive evaluation of the specimens' performance under cyclic thermal stress.

2.3.8 Test Monitoring and Data Collection

Throughout the cyclic thermal test, researchers diligently monitor and systematically record relevant parameters. These parameters include temperature, time, and any observations or measurements of the specimens' response to thermal stress. This meticulous data collection is crucial for evaluating the specimens' performance and behaviour under cyclic thermal conditions, providing valuable insights into their high-temperature oxidation resistance.

2.3.9 Heating and Cooling Cycles

Specific time durations and temperature levels are determined for the heating and cooling cycles based on testing requirements and the material being evaluated. For instance, the heating cycle involves increasing the temperature to 600°C and maintaining it for 5 hours. The subsequent cooling cycle entails reducing the temperature from 600°C back to ambient conditions over a 24-hour period.

2.3.10 Dwell Periods and Hold Times

While the provided information does not explicitly detail specific dwell periods or hold times at temperatures, researchers may strategically introduce optional dwell periods during the heating or cooling cycles. These intervals, if included, serve purposes such as stabilization or observation at specific temperatures, providing a more nuanced understanding of the specimens' behaviour under prolonged exposure to specific conditions. The decision to include dwell periods is contingent upon testing requirements, material properties, and the specific insights sought from the cyclic thermal test, offering adaptability to unique material characteristics and testing objectives.

3.0 Result and Discussion

3.1 Microstructure

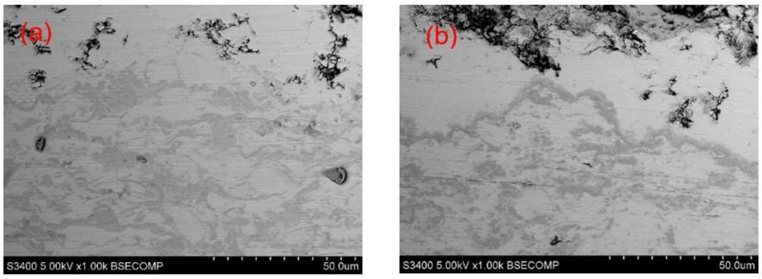

Figure 3: Microstructure of sample (a) Lanthanum Zirconia/ Yittria Stabilized Zirconate (b) Yttria Stabilized Zirconate.

The analysis of the figure 3 sheds light on the oxidation behavior of dual-layer (DCL) and single-layer (SLC) coatings after undergoing a 90-hour high-temperature oxidation test. Notably, the DCL coating exhibits superior oxidation resistance compared to the SLC, evident from the absence of a black area representing the Thermally Grown Oxide (TGO) layer. However, within the DCL coating, a grey area indicates the presence of mixed oxides, including chromia, spinel, and nickel oxides (CSN), suggesting a complex but effective defense against oxidation. In contrast, the single-layer YSZ coating shows a clear TGO line after 90 hours, indicating a less robust oxidation resistance compared to DCL. The cross-sectional morphology of the YSZ coating reveals a continuous Al_2O_3 scale (black area), distinct from DCL, 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, emphasizing the influential role of layer thickness ratios in determining the overall oxidation resistance of the coatings.

3.2 EDX

3.2.1 10 Hours

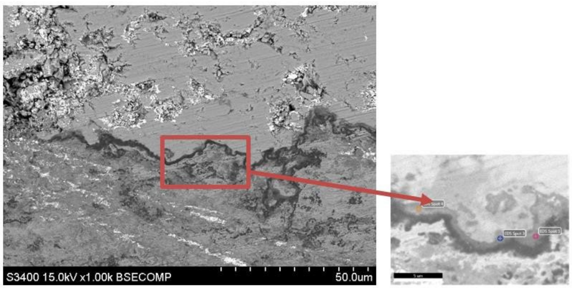

Figure 4: Close up TGO layer for YSZ.

Table 1: Data of Elements in TGO layer YSZ

Coloured	Element	%Atomic
Dark Area	Al_2	20.42
	0_3	42.68
Gray Area	Cr	17.92

	Co	8.36
	Ni	10.62

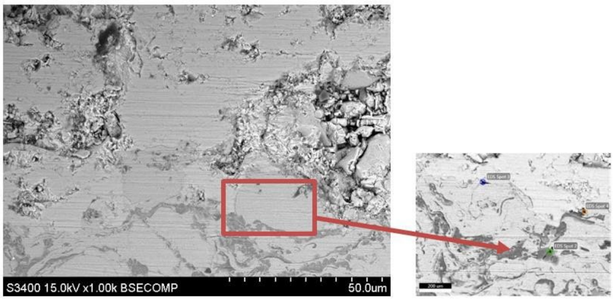

Figure 1: Close up TGO layer for LZ/YSZ

Table 2: Data of elements in TGO layer LZ/YSZ

Coloured	Element	%Atomic
Dark Area	Al_2	30.5
	0_3	28.98
Gray Area	Cr	14.55
	Co	14.66
	Ni	11.31

3.2.2 90 Hours

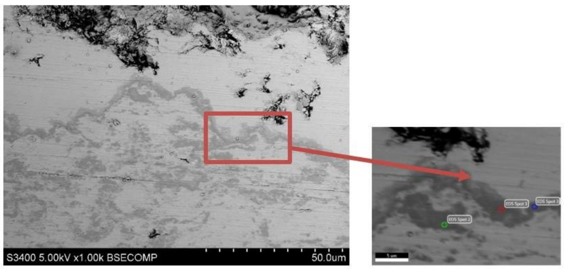

Figure 2: Close up TGO layer YSZ.

Table 31: Data of Elements in TGO layer of YSZ

Coloured	Element	%Atomic
Dark Area	Al_2	18.77
	0_3	31.98

Gray Area	Cr	19.01
	Co	14.3
	Ni	15.94

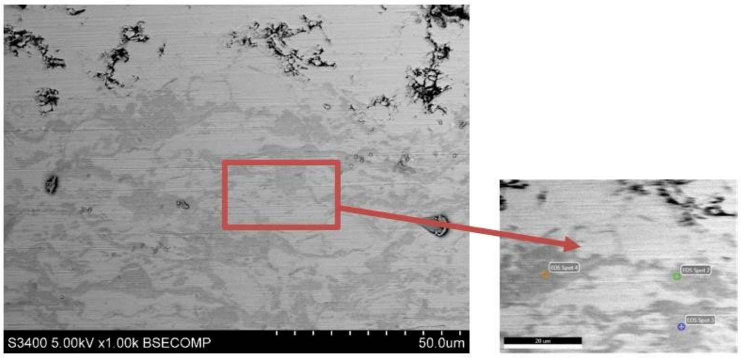

Figure 3: Close up TGO layer for LZ/YSZ

Table 4: Data of elements in TGO layer of LZ/YSZ

Coloured	Element	%Atomic
Dark Area	Al_2	20.55
	0_3	30.28
Gray Area	Cr	16.55
	Co	14.84
	Ni	17.78

The elemental composition analysis of the coated sample at 10 and 90 hours reveals notable changes in both the dark and grey areas, offering insights into material transformation over time. In the dark area, representing the primary coating, there is a decrease in the percentage of Al_2O_3 from 20.42% to 18.77%, and O_3 from 42.68% to 31.98% between 10 and 90 hours. This suggests a reduction in Al_2O_3 content and potential alterations in the oxide composition, indicating ongoing oxidation processes.

In the grey area, encompassing mixed oxides like Cr, Co, and Ni, significant changes are observed. The percentage of Cr increased from 17.92% to 19.01%, Co from 8.36% to 14.3%, and Ni from 10.62% to 15.94% over the same period. These variations indicate a dynamic evolution in the composition of mixed oxides, likely due to oxidation and transformation processes during extended heat treatment.

The increased percentages of Cr, Co, and Ni underscore the importance of these elements in the evolving composition of the grey area. In summary, this elemental composition analysis provides valuable insights into material changes during high-temperature oxidation testing, enhancing our understanding of the coating's behaviour over time.

3.3 Weigh Gain

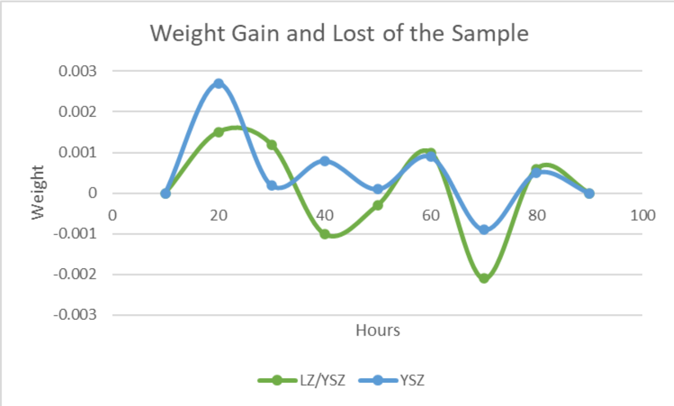

Figure 8: Graph represent Weight Gain and Lost of Sample

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.

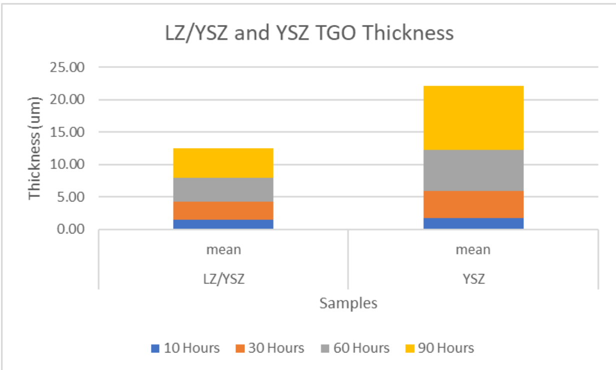

Figure 9: Comparative Analysis of LZ/YSZ and YSZ Samples by Layer Thickness in Micrometres (µm) with Mean Values

The provided graph is a stacked bar chart comparing the thickness of thermal grown oxide (TGO) layers in two samples: LZ/YSZ and YSZ. The thickness of the TGO layers is presented as a mean value in micrometres (µm), segmented into four time-based categories represented by different colours: blue for 10 hours, orange for 30 hours, grey for 60 hours, and yellow for 90 hours. This indicates that the measurements were taken after these specific durations of oxidation. The graph shows that the TGO layer thickness increases with time for both samples, which is consistent with the expected behaviour of TGO layers growing because of oxidation at elevated temperatures.

Analysing the graph, it is evident that the YSZ sample has a significantly greater mean thickness across all time intervals compared to the LZ/YSZ sample. Specifically, after 90 hours of oxidation (the yellow segment), the YSZ sample shows the greatest increase in thickness. This suggests that the YSZ material undergoes more substantial oxidation over time, which could imply a faster oxide growth rate, or a different oxidation mechanism compared to the LZ/YSZ sample. Such data is crucial in high-temperature oxidation testing, as the growth rate of TGO can impact the performance and lifespan of protective coatings in turbines, engines, and other thermal barrier systems.

4.0 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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