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Thermochemical and Thermomechanical Study on Composite Coatings (Al_2O_3+ZrO_2·5CaO)

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***Abstract*: Premature failure is the major apprehension in graded coating systems. To determine the life span of the topcoat (Al_2O_3+ZrO_2·5CaO), Muffle furnace technique was adopted. The topcoat thicknesses were varied in 100, 200 & 300 µm and applied on Cast iron substrate. A controlled heating (600±2°C) and ambient cooling in the cycle were carried out until the fracture acknowledges at the surface of the topcoat. Results obtained from SEM micrograph and EDX analysis revealed that thermochemical and thermomechanical stresses lead to premature failure of the topcoat. The coating reported failing from the bond coat and the same is remained intact with the substrate. After 312 thermal cyclic tests, spallation acknowledges in the case of 100 µm topcoat. The mechanism of failure for the above said graded composite coating discussed in detail.**

***Index Terms*: Al_2O_3+ZrO_2 .5CaO; Muffle furnace; Thermochemical; Thermomechanical.**

I. INTRODUCTION

Generally, Thermal Barrier Coatings (TBC) are developed based on the required applications. The choice of compatible elements in the graded coating systems plays a major role in determining the life span of the coatings. Among several parameters viz. material preparation, initial surface treatment, & coating methodology etc., the coefficient of thermal expansion (CTE) has a significant contribution in the determination of TBC life. It is often recommended to develop a composite mixture with comparable chemistry and affinity [1]. Several TBC failure reported due to inconsistencies in the topcoat and bond coat elemental compositions in the graded coating systems [2]. It has been reported that topcoat fails when Al2O3 scale reaches to its critical thickness, formed mainly at the interface of the topcoat and bond coat [3]. The crack initiation has been also, acknowledged due to thermal mismatch at the interfaces of topcoat/TGO/bond coat in the case of spinel oxides and Al_2O_3[4].

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Most of the earlier studies focused on the crack growth of the Al_2O_3 in TBC's and conducted the experiment in the temperature range of 1000 to 1200 °C and volume expansion allied with phase conversion reported at 1170 ºC[5-9]. In this paper, the life span of the composite coatings (Al_2O_3+ZrO_2·5CaO) were determined using a muffle furnace test rig. The heating and cooling cycle was maintained for 30 minutes. A controlled temperature 600±2 °C set for heating and ambient cooling was done. Sufficient discussion made on the thermochemical and thermomechanical mode of failure mechanism for the Alumina and Calcia stabilized zirconia graded composite coating.

II. EXPERIMENTAL METHODOLOGY

A. Selection of material and powder trade names

Trade Name	Composition by wt.%
Metco 105 SFP	99.9% Al_2O_3
Metco 201 NS	ZrO_2.5CaO
Metco 452	Fe38Ni10Al

Fig. 1. Trade name and composition of the powder

Cast Iron (CI) was selected as a substrate material. The selection of powder was based on the thermal coefficient of provided by Sulzer Metco.The trade names of different powders are shown in Fig. 1.

B. Coating Methodology

Top Coat (100 µm) Pure Ceramic (ZrO_2.5CaO+Al_2O_3)
Bond Coat (50 µm) (Metallic Powder) Fe38Ni10Al
Substrate (Gray Cast Iron)

Fig. 2. The schematic of coating system applied on cast iron

The Atmospheric plasma spray technique was used to coat on the cast iron substrate. Before the coating process, the mixture of Al_2O_3 and ZrO_2·5CaO in 50:50 was prepared using ball mill technique. The substrates were chemically cleaned using tetra chloride-ethylene followed by preheating it to the temperature of 250±50 ºC.

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This process was done to minimize the thermal mismatch between the substrate and the bond coat. The schematic of a coating system is shown in Fig. 2.The top coat thicknesses were about 100µm.The plasma spray machine specification and spray parameters for bond coat and top coat are given in Fig. 3 and 4 respectively.

Specifications	Parameters
Plasma gun	3 Nylon Brush
Nozzle temperature	10,000 °C
Current	500 amps
Voltage	65-70 volts
Powder feed	45-50 gms/mint
Spray distance	50 -78 mm

Fig. 3. Air Plasma machine specification

Materials	Primary gas (Argon) Pressure(Bar)	Secondary gas (Hydrogen) Pressure (Bar)	Carrier gas Argon flow (lpm)	Current (amps)	Voltage (volts)	Spray distance (mm)
Al_2O_3+ ZrO_2.5CaO	3.7	3.45	35	500	65	65-76
Fe38Ni10Al	6.9	3.30	35	500	65	50-76

Fig. 4. Plasma spray parameters for different coating materials.

C. Thermal cyclic test procedure

The thermal cyclic test was conducted on a Muffle furnace. The schematic of the test setup shown in Fig.5. The temperature of the furnace was maintained at a temperature of 600±2ºC. The coated substrate was kept on silica ceramic cups to ensure uniform heating all-around refer Fig.5. The heating and cooling cycle time of 30 minutes was maintained throughout the experiment. The cooling was done under ambient conditions.

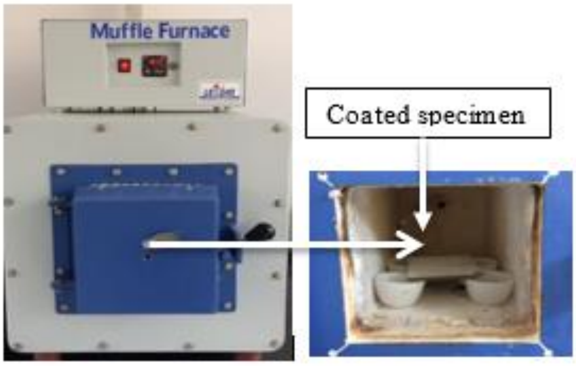

Fig. 5. Muffle Furnace test setup

D. Coating Characterization

Evaluation of the coating thickness, surface morphology, and determination of elemental composition was carried out using Zeiss Evo 18 special edition machine, and the machine specifications are given in Fig. 6. XRD analysis was carried out on Bruker, and its specifications are given in Fig. 7.

Filament	Tungsten
Secondary e-image resolution	50 NM
Tilt	0 - 60 Degree
Rotation	360 Degree
EHT	200V - 30KV
Magnification	Up to 50K ~ 100K (Depends on sample)

Fig. 6. Machine Specifications of Zeiss Evo 18

Parameters	
Make	Bruker
Model	D8 Advance
Measuring circle diameter	435,500,600
Smallest addressable increment	0.0001°
Reproducibility	0.0001°
Anode	Cu
Detector	Scintillation & Lynx eye

Fig. 7. X-Ray Diffractometer Specifications

III. RESULT AND DISCUSSIONS

After 295 cycles oxidation mark acknowledges on the topcoat [Fig. 8(a)]. Among all the coating systems (100, 200, & 300 µm), 100 µm topcoat ($ZrO_2\cdot5CaO$+Al_2O_3) found to be fractured after 312 thermal cycles [Fig.8 (b)].The two primary reason of failure noticed and elucidated, namely failure due to thermomechanical effect (stress inversion) at the interface of the top/bond coat and the failure due to thermochemical effect (oxidation of the bond coat). Both the modes of failure have been discussed in detail.

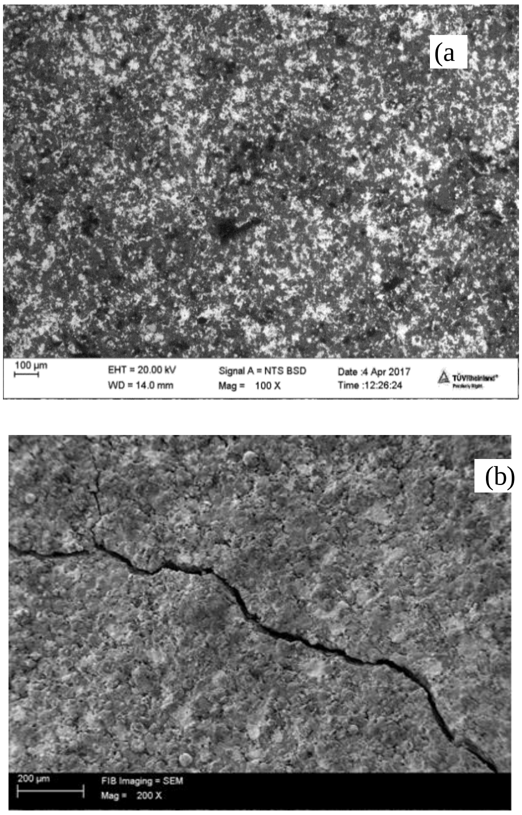

Fig. 8. SEM Micrograph (a) oxidized status after 295 cycle (b) crack after 312 thermal cycles test

A. TBC failure due to Thermomechanical effect

The coating failure mechanism can be understood from stress inversion theory. The root cause of crack nucleation and propagation has been studied and established earlier also and has been described on the simplified wave trend profile. The wave trend describes the coating stress states under thermal and mechanical loading system [10-12].

Fig .9(a) and (b) shows the SEM micrograph without and with TGO at the cross-section of the 100 μm coating system. Fig.10 (a) shows the as-sprayed coating interface state of the topcoat and bond coat. From the stress inversion theory in case of the as-sprayed coating without TGO, the stress state may be understood as follows: the tensile stress exists in the crest zone and compressive stress exists in the trough zone at the interface of the coating system. However, the growth of TGO, during thermal cyclic test led the inversion of stress state and is shown in the Fig.10 (b). The inversion of stress state may be attributed to mismatch in the thermal coefficient of expansion between top coat and TGO and between TGO and bond coat also, under the influence of elevated temperature and thermal cyclic loading crack disseminate from the crest along the slope till it reaches to trough zone and eventually spallation of the topcoat due to difference in the magnitude of stress state. The thermal coefficient of expansion of the topcoat and bond coat, topcoat and bond coat surface anchoring (asperities-stressed zones), porosity and loading conditions significantly influence the life of the coating system [13-17].

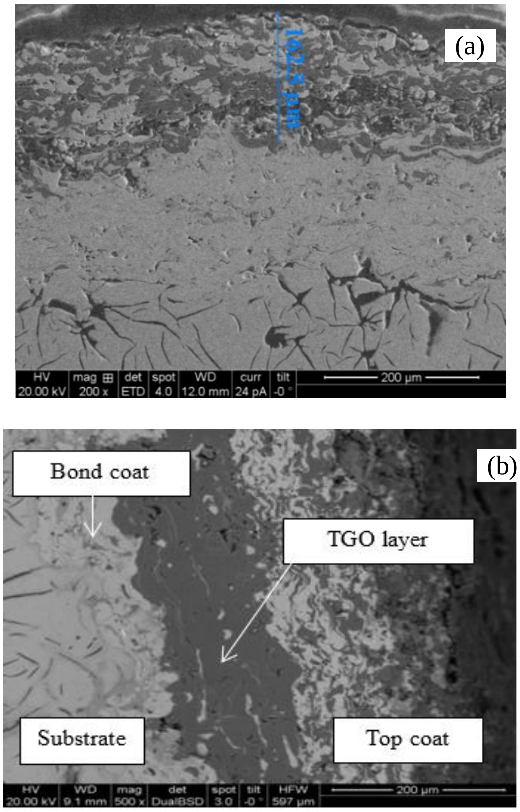

Fig. 9. SEM Micrograph (a) Without TGO (b) With TGO

B. TBC failure due to Thermochemical effect

Lager proportion of iron content (Fe) i.e. 52 wt. % and differential porosity on the topcoat and bond coat led thermal instability in the graded coating system. The average porosity for as-sprayed coating in case of the topcoat and bottom coat found to be 1.90 & 2.75%, [Fig. 11 (a) & (b)] respectively. Porosity considered as one of the important factors in the determination of the life span of the coatings [18].The trace amount of Fe element, 1.51 wt. % along with oxygenated element 50.85 wt. % has been also confirmed by EDX analysis. Fig. 12 (a) and (b) shows the elemental present on the topcoat of as-sprayed coatings and after 312 thermal cyclic tests. The thermochemical reaction triggered beneath the topcoat i.e. when Fe in the bond coat (Fe38Ni10Al) exposed to the ambient cooling in presence of moisture, redox reaction initiated and led the oxidant product appears on the topcoat during thermal cyclic test and the same can be understood with the following (1).

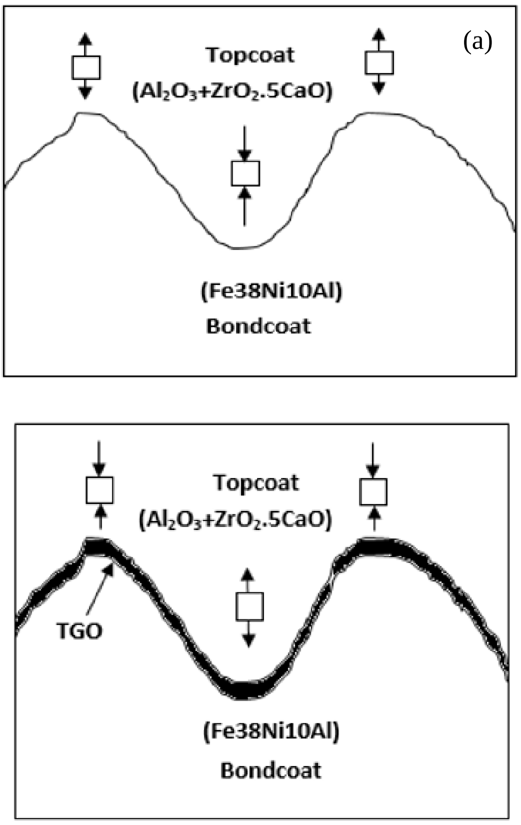

Fig. 10. (a) As sprayed without TGO coating interface state (b) With TGO coating interface state

$$Fe^{2+} + O_2 + \Delta \rightarrow 4\ Fe^{3+} + 2\ O^{2-} \qquad (1)$$

During the test, it was noticed that the thermally grown oxide layer (TGO) developed at the junction of the bond coat and top coat resulting weakening of the top coat/bond coat interface [Fig. 9(b)]. The growth of the TGO layer found to increase with passes of time. Spallation of the topcoat from the bond coat also acknowledged in the work carried out by Julian D. Osorio [19].

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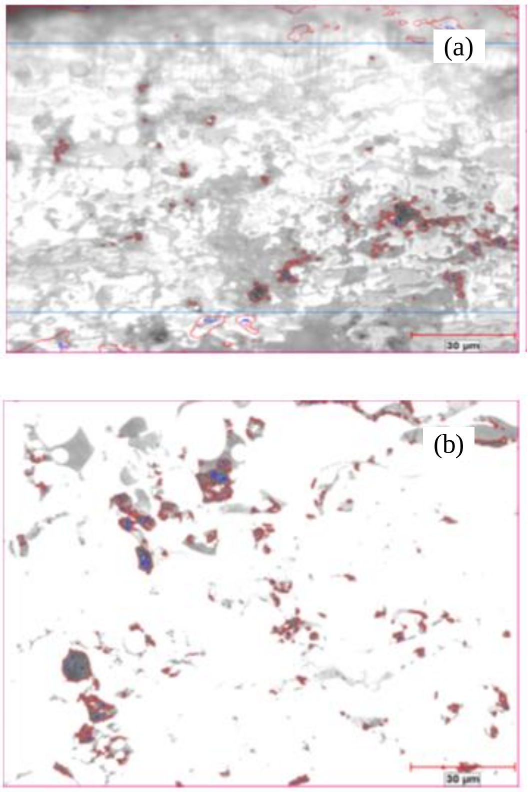

Fig. 11. Micrograph of as-sprayed micro porosity of (a) topcoat 1.90 % and (b) bond coat 2.75%.

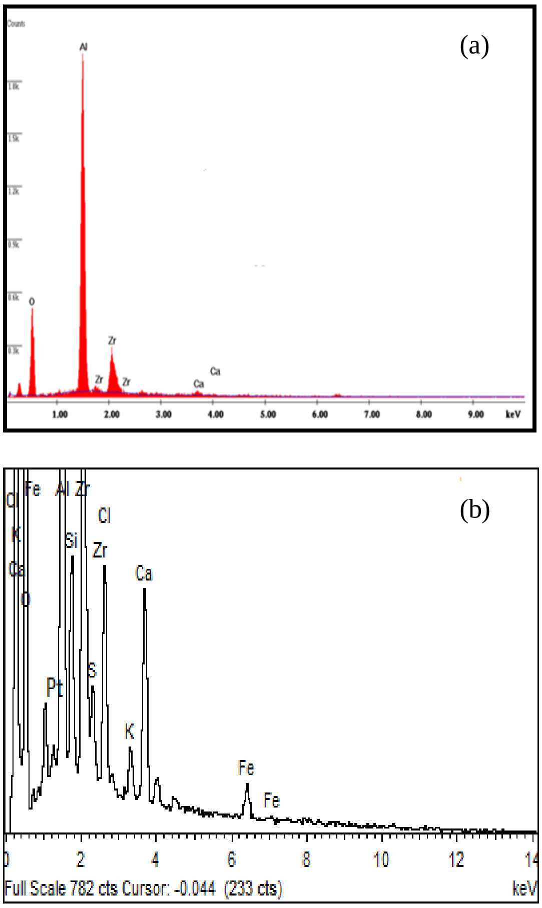

Fig. 12 Elemental composition/spectrum of (a) as-sprayed topcoat and (b) after 312 cycles top coat

IV. CONCLUSION

From the muffle furnace thermal cyclic study following conclusion can be made:

1. Initial crack acknowledged after 312 thermal cyclic tests iN the case of 100µm topcoat (ZrO_2.5CaO+Al_2O_3). Improved coating life observed at higher coating thickness.

2. Thermochemical, (oxidation of Fe element) and Thermomechanical (Stress inversion) are the two failure modes mainly observed in the present coating systems.

3 Micro porosity and a relatively high weight percent of Iron (Fe) element in the bond coat (Fe38Ni10Al) and relatively less coating thickness (100µm) led the top coat delaminated from the bond coat.

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