

IMPROVED OXIDATION RESISTANCE FOR THERMAL BARRIER CERAMIC COATING PROTECT

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ABSTRACT

The oxidation resistance of Ni base super alloy (Monel 400) can be improved by thermal sprayed coatings produced by the high velocity oxygen fuel (HVOF). In this work, (Monel 400) is coated with two different types of coatings, the first one is $\text{Al}_2\text{O}_3+5\% \text{TiO}_2$ and the second is $\text{Al}_2\text{O}_3 +15\% (7-8\text{YSZ})$, these layers were made of 350-400 μm as top coat, pre-sprayed with 50-100 μm of 4NiCr5Al as a bond coat. The results revealed that the $\text{Al}_2\text{O}_3+5\% \text{TiO}_2$ have higher oxidation resistance than $\text{Al}_2\text{O}_3 +15\% (7-8\text{YSZ})$ in the temperature range (600-800)⁰C in air for 15 h at 3h cycle. X-Ray diffraction (XRD) was used to identify phase formed in the surface layer of as-coated specimens before and after oxidation test. Phase transformation is accompanied by volume expansion, leading to compressive stress during oxidation. It is mentionable that the compressive stresses lead to increase of the hardness. Hardness of $\text{Al}_2\text{O}_3 +15\% (7-8\text{YSZ})$ at (600-700 and 800)⁰C is (723,765 and 812) $\text{HV}_{0.3}$ respectively is more than $\text{Al}_2\text{O}_3 +5\% \text{TiO}_2$ at the same conditions (610,645 and 690) $\text{HV}_{0.3}$ respectively.

Porosity transferred oxygen from top coat layer toward the bond coat (4NiCr5Al). Hence, a thermally grown oxide layer () was formed on the metallic bond coat and internal oxidation of the bond coat occurred during oxidation. Microstructural characterization of coatings demonstrated that the growth of the TGO layer with $\text{Al}_2\text{O}_3+15\% \text{YSZ}$ is more rapid than TGO with $\text{Al}_2\text{O}_3+5\% \text{TiO}_2$. To assess Adherence Index, porosity and the hardness for the $\text{Al}_2\text{O}_3 +5\% \text{TiO}_2$ coating are 84%, 9% and 454 $\text{HV}_{0.3}$ respectively while for the $\text{Al}_2\text{O}_3 +15\% (7-8\text{YSZ})$ coating are 80.1, 17% and 483 $\text{HV}_{0.3}$ respectively.

KEYWORDS: Sprayed Coatings, Oxidation Resistance, Alumina - Titania, Alumina - Zirconia

INTRODUCTION

Ni-base superalloys are the commercial alloys commonly used for high-temperature applications, e.g. manufacture of components used in aggressive environments of gas turbines, steam boilers, etc. Ni-base superalloys (Monel400) corrosion resistance is very good in reducing environments, but poor in oxidizing conditions [1]. Extreme environments can result in the loss of material to oxidation/ corrosion process along with decrease in mechanical properties of the substrate due to deleterious elements diffusing into the substrate at high temperatures. In order to meet the challenge of the rising cost of high performance materials. Surface coating techniques have recently attracted the attention of investigators worldwide [2].

Thermal barrier coatings (TBCs) are widely used for this applications. The TBCs are usually composed of a MCrAlY bond coat (M = Ni, Co) as an oxidation resistant layer also in order to enhance the bond strength and reduce the effect of thermal expansion coefficient mismatch between the top coat and the substrate alloy, and yttria-stabilized zirconia (YSZ), Al_2O_3 and TiO_2 as a topcoat that provides thermal insulation toward metallic substrate [3, 4].

The selection of thermal barrier coating materials is restricted by some basic requirements. These requirements are high melting point, no phase transformation between room temperature and operating temperature, low thermal conductivity, chemical inertness, thermal expansions match with the metallic substrate, good adherence to the metallic substrate and low sintering rate of the porous microstructure. So far, only a few materials have been found to basically satisfy these requirements [5].

Although much progress has been made in the development of new materials, there exists at present no single material which can withstand all the extreme operating conditions in modern technology. So that the combined properties of the composite system can satisfy a particular set of operating conditions [6].

Coatings based on alumina are a good alternative when wear resistance and chemical stability are the controlling factor. Alumina is hard but its main drawback is its brittleness [7, 8]. The addition of titanium oxide leads to a balanced equilibrium of properties maintaining enough hardness and increasing considerably the coating toughness. Titanium oxide has a lower melting point and plays a role of binding alumina grains to achieve coatings with a higher density and wear resistance coating.

Zirconia shows only 50% of alumina's hardness but transformation toughening improves fracture resistance, that is overall toughness and bending strength are substantially higher than for alumina [9] Al_2O_3 is not soluble within ZrO_2 and forms a rigid matrix around the ZrO_2 particles which causes a local compressive stress and hinders the phase transformation of zirconia [10].

These coatings are usually applied by a thermal spray process because a very high temperature is required to melt the ceramic powder. Coatings produced by HVOF spray have comparatively lower porosity, higher hardness and superior bond strength than many of the other thermal spraying methods such as plasma spraying [11].

K. N. Lee, [12] 2000

Identified the major issues related to the selection of TBC systems. He indicates that the coating should have the ability to resist reaction with the aggressive environment, with low oxygen permeability. In addition, the coefficient of thermal expansion of the coating should be close to that of the substrate material along with their chemical compatibility. An additional desired quality is their ability to maintain a stable phase in a thermal environment.

A. C. Karaoglanli et al. [13] (2011)

Investigated that adding alumina (35 wt. %) into the YSZ is effective in improving the thermal cycle life time of YSZ (yttria stabilized zirconia) thermal barrier coatings. However crack propagation during thermal cycling appears to be controlled by TGO growth. Alumina particle composited top coatings tend to have low oxygen diffusion. This is helpful to limit thermal grown oxide on bond coat and improve the thermal cycle life time and mechanical properties of YSZ top coat at 1200 °C.

Sharafat et al. [14] 2000

Developed a novel $\text{ZrO}_2\text{-Al}_2\text{O}_3$ thermal barrier composite coating by using the gas tunnel type plasma spraying and the best composite microstructure was found for 50% Al_2O_3 powder mixture. - *Limarga et al. [15] 2002*: studied the oxidation behavior of duplex and functionally graded $\text{Al}_2\text{O}_3/\text{ZrO}_2$ TBCs by examining the microstructure in the as-sprayed specimens; they conclude that Al_2O_3 is effective as an oxygen barrier layer, minimizing the delamination of the coating of as-sprayed components. In addition, the interlayer also reduces the interfacial residual stress induced by TGO after

being exposed to high temperature environment. Also the effects of phase transformation on the structural integrity are minimized by applying thinner Al_2O_3 .

A. Afrasiabi et al. [16] 2008

Investigated that formation of monoclinic ZrO_2 was suppressed in (YSZ / Al_2O_3) than usual YSZ, because the presence of fine-grained Al_2O_3 around YSZ particles reduced direct contact of YSZ with molten salt. Also the researcher investigated in(2013)[17] the Monoclinic ZrO_2 and YVO_4 crystals as hot corrosion products caused substantial compressive stresses in usual TBC which led to increase of hardness and elastic modulus in the YSZ and resulted in the degradation of the coating, while these stresses decreased in the YSZ/ Al_2O_3 system.

C. Zhu et al. [18] 2012

Investigated that the existence of Al_2O_3 in YSZ is beneficial to stabilize the tetragonal prime phase (t' -phase) during oxidation. During cyclic oxidation at 1000 °C for 40 h, the weight gain per unit area of Al_2O_3 /YSZ TBCs was smaller than that of as-sprayed YSZ TBCs.

A. Rico et al. [19] 2010

Studied isothermal oxidation test at 400, 500, 600 °C performed on Al_2O_3 -13% TiO_2 (wt. %) coatings deposited on SAE- 42 steel by atmospheric plasma sprayed (with using Ni-Al-Mo 90/5/5 (wt. %) as bond coat), he investigated that the oxidation products and the scales morphology depend on oxidation temperature and time. Two oxidation layers can be observed in the environmental barrier system. The first one grows between the ceramic top coat and the bond coat; and the main corrosion products are α - Al_2O_3 and NiAl_2O_4 . The second layer is located at the interface between the bond coat and the substrate and it is mainly formed by the NiFe_2O_4 spinel. Even FeO can be finally obtained.

K. A. Habib et al. [20] 2011

Studied the oxidation behavior of alumina/titania (97/3, 87/13 and 60/40) ceramic coatings, both layers were deposited on an AISI 304 stainless steel base metal by the flame spray technique. The coated steel was heated from room temperature to 1, 123 K, oxidized in air for 50 h, and then cooled to room temperature. Coatings 97/3 and 87/13 presented a stable structure after flame spray deposition and they did not evolve with the oxidation process, while most of the 60/40 coating changed to a metastable structure after deposition and to a more stable structure after oxidation with high micro-cracks content.

In this work, the oxidation behavior of Al_2O_3 +5% TiO_2 and Al_2O_3 +15% YSZ during the exposition of the coatings to an air environment has been studied. Oxidation products (phases) have been identified and its influence on the coatings behavior has been established.

EXPERIMENTAL WORK

Deposition of Coatings

The substrate material selected for the present study was a Nickel based superalloy (Monel 400) this alloy finds applications in boiler components. The chemical composition of the substrate material is presented in table 1. Specimens with dimensions of approximately (10mm X 10mm X10mm, they were cut from a plate to the required shape. The samples were prepared for spraying process by grit blasting and cleaning before applying coating material.

Table 1: Chemical Composition of Ni Base Supper Alloy (Monel 400)

Elements Materials	Ni %	Cu %	Fe %	Mn %	C %	Si %	S %	Other
Nominal Chemical Composition [21]	63.0 min	28.0-34.0	2.5	0.20	0.3	0.5	0.024	
Actual Chemical Composition	64.4	32.61	2.44	0.189	0.234	0.481	0.018	

Four types of commercial powders were selected: 4Ni Cr 5Al with (50-90 μ m) as bond coat, an 8% Yttria-Stabilized Zirconia (YSZ) type (Saint-Gobain USA), TiO₂ type (GCC England), Al₂O₃ type (Panreac Spain) has been used for TBC coating. In order to produce particle composite, two types of coatings were produced by the high velocity oxygen fuel (HVOF) method which included: Alumina (Al₂O₃) + 15% Yttria Stabilizer Zirconia (7-8YSZ), Alumina (Al₂O₃) + 5% Titania (TiO₂).as top coat. Each type of this powders were sieved in range 10-45 μ m by using a sieve analyzer with microsieves. Two batches with different mixing ratio (wt/wt) were prepared. The selected ratio of alumina-titania powders were mixed in the laboratory ball mill for 4 h, also for alumina -zirconia to get a uniform mixture of powders.

The coating technique in this work is the flame spray method type (rototec 800). This apparatus consists of a chamber containing a flange to hold the specimen and an Oxy- Acetylene flame. The powder particles flow with the flame and is deposited on the specimen. The powder was supplied through a special tube in the flame gun. The surface of metal was cleaned and roughened using emery paper (p220) and grit-blasted using sand blast system with pressure (4-6) bar. Then, the grit-blasted substrates were cleaned using anhydrous ethanol alcohol and dried to 200 °C by a furnace for 30 min. The ceramic powder Al₂O₃ +5% TiO₂and /Al₂O₃+15% YSZ with particle sizes 10-45 μ m and NiCrAl metal powder (bond coat) with particle sizes ranging from 50 to 90 μ m were used. The specimen is fixed on the flange to make 90° with the powder flame flow. Then, the cooling system (air compressor) is switched on to cool the specimen from behind and keep it from melting during spraying process. The system is switched on and the flame is ignited. The flame holder is controlled manually. The bond powder required for the first layer is loaded into the hopper, and the specimen is heated to a suitable temperature around (300 °C) by the flame. The coating process is started by moving a lever on the hopper to allow all the powder to flow through the hopper with the flame with a distance of about (20 cm) between the flame and the specimen. The previous step is repeated until 50 to 100 μ m thickness of bond layer has been reached. The ceramic powder (required for the top coat) is then loaded and the same procedure of bond was also repeated until 350 to 400 μ m thickness has been reached. The coating is heated to about 1500 °C for a suitable time to permit the adhesion for layers. The temperature for bond coat and top coat is controlled by adjusting the distance between the flame and the specimen and the pressure of Oxy-Acetylene (about 1500 °C for bond layers and 2800 °C for the ceramic layers). The flame is withdrawn gradually away from the specimen to minimize thermal shock. To measure the thickness of coating layer after spraying process, Coating thickness gauge type (QuaNix1500 Germany) was used, Infrared thermometer was used to know the temperature of surface before spraying process and the temperature of the fusion after spraying process

Characterization of As-Sprayed Coating

In accordance with EN10209, the test in European standard of adherence strength, the coated specimen was tested by the steel ball falling impact. The adhesion strength was judged according the relics of coating on the destroyed surface. For obtaining a percentage number for adherence index, the imaging of microstructure of the peeled surface after the adherence strength test for samples was done by using an optical microscope with a digital camera.

Then, the microstructure images were processed using "Adobe Photoshop CS6" program and the adherence index was calculated using the "Image J" program.

The as-sprayed coating was examined porosity, porosity measurements were carried out using boiling water immersion method for open pores determination. The procedure is summarized as follows:

- Determination of the dry weight (W) in grams.
- Immersion the samples in distilled water and boiling for 2 hours.
- After cooling to room temperature, weighting the suspended sample in distilled water (W_s) in grams.
- Determination the weight of saturated specimen with water in air (W_p), after removing all drops of water from the surface.

From above procedure the following parameters can be obtained

$$\text{Apparent porosity (P\%)} = \frac{\text{Volume of Open Pores}}{\text{Volume of Specimen}} \times 100 \quad (1)$$

$$\text{The volume of specimen} = W - W_s \text{ in } cm^3 \quad (2)$$

$$\text{The volume of open pores} = W - W_p \text{ in } cm^3 \quad (3)$$

X-ray diffraction (XRD) analysis of the coated samples before and after oxidation test was carried out using a Riga X-ray generator with Cu K_α radiation at 60Kv and 80mA, The X-ray is generated by general electric diffractometer, type Shimadzu X-Ray diffractometer (6000/7000), Japan. To detect any crystalline zone in the specimens surface.

High-Temperature Oxidation Studies

Ni base super alloy (Monel400) with and without coating samples were accurately weighed and then suspended with platinum wire into ceramic crucibles. The specimens were set in an electric furnace with air atmosphere at 600-700-800 °C for 15 h and then cooled down inside the furnace. The specimens were inspected periodically after each of 3 h.

Samples of weight changing before and after each oxidation cycle were measured. Normally: at least 3 weight measurements were taken. Weight was taken with help of sensitive Electronic Balance with a sensitivity of 0.0001 g.

RESULTS AND DISCUSSIONS

Characterization of As-Sprayed Coating

Alumina titania and alumina zirconia specimens have different in some properties as shown in table

Table 2: Illustrate Comparison between $Al_2O_3+5\% TiO_2$ and $Al_2O_3+15\%$ (7-8YSZ) Coating Properties

Coating Materials Properties	$Al_2O_3+15\%$ (7-8YSZ)	$Al_2O_3+5\% TiO_2$
Hardness (HV) _{0.3}	483	454
Porosity %	17	9
Adherence Index (%)	80.1	84

Oxidation Test

Figure 1, 2 and 3 shows the oxidation kinetic curves with AT, AZ coating and without coating specimens at 600, 700 and 800°C. The mass gain was quite similar to each other till 6 h with a fast increase since that especially at 800°C. As can be seen the AT and AZ coating showed lower oxidation rate than that of without coating specimen. The distinction of the mass gain between the two coatings increased with extending exposure time.

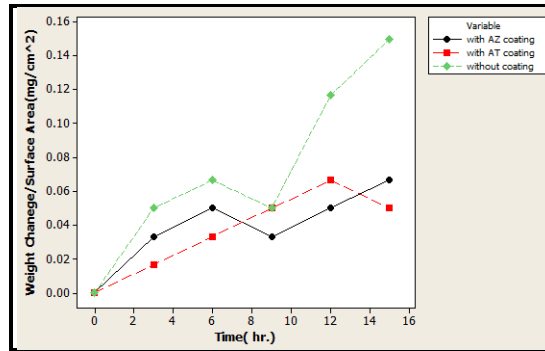


Figure 1: Weight Change/Surface Area (Mg/Cm²) vs. Time Plot for Uncoated Ni Base Supper Alloy (Alloy 400), AT and AZ Cyclic Oxidized in Air at Temperature 600°C for 15hr at 3hr Cycle

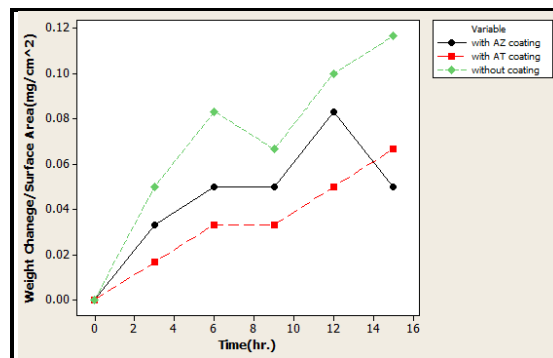


Figure 2: Weight Change/Surface Area (Mg/Cm²) vs Time Plot for Uncoated Ni Base Supper Alloy (Alloy 400), AT and AZ Cyclic Oxidized in Air at Temperature 700°C for 15hr at 3hr Cycle

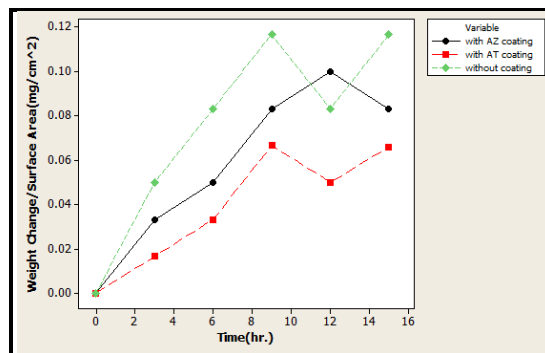


Figure 3: Weight Change/Surface Area (Mg/Cm²) vs Time Plot for Uncoated Ni Base Supper Alloy (Alloy 400), AT and AZ Cyclic Oxidized in Air at Temperature 800°C for 15hr at 3hr Cycle

X-Ray Diffraction

The coating was characterized by X-ray diffraction Figure 4 a illustrates the XRD patterns of as sprayed Al₂O₃+15% YSZ that include the tetragonal phase of ZrO₂, rhombohedra (α) phase of Al₂O₃. Figure 4 b, c, d exhibits the XRD patterns of the coatings after oxidation process. Monoclinic and tetragonal phases of ZrO₂ and tetragonal (δ) phase Al₂O₃.

While Figure 5a illustrates the XRD patterns of as sprayed $Al_2O_3+5\%TiO_2$ that include the rhombohedra (α) phase of Al_2O_3 and TiO_2 . The partially disappearance of tetragonal(δ)as compared to figure 4b, c, d and the typical peaks of Rhombohedra (α), cubic (γ) stable alumina were detected after the oxidation process also found TiO_2

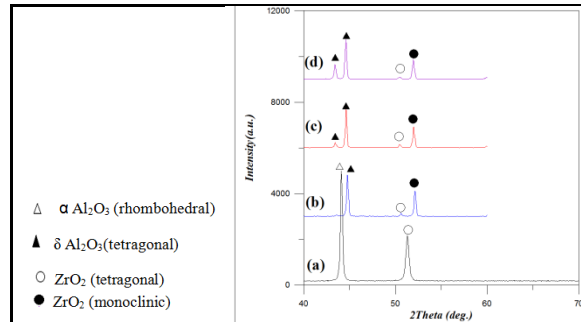


Figure 4: X - Ray Pattern of AZ A: As Sprayed, B: At 600°C, C: at 700°C, D: at 800°C

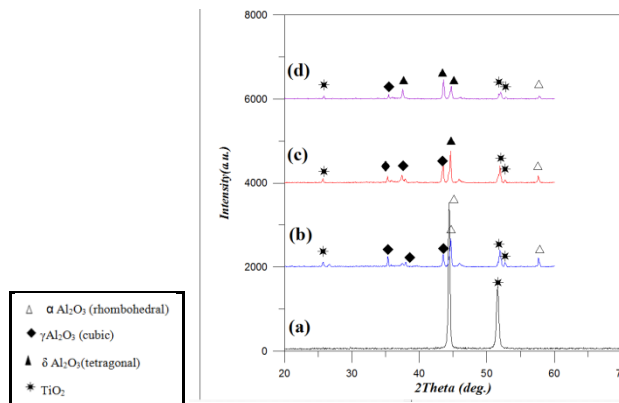


Figure 5: X - Ray Pattern of AT A: As Sprayed, B: at 600°C, C: at 700°C, D: at 800°C

Oxidation Test Effect on Coating Layer Microhardness

The increased hardness values are related to the phase transformation is accompanied by volume expansion, leading to compressive stress during oxidation. It is mentionable that the compressive stresses lead to increase of the hardness, α and γ - stable phases of Al_2O_3 were detected after the oxidation process and the partially disappearance of tetragonal (δ) in AT specimens.

While in AZ specimens found tetragonal (δ) phase Al_2O_3 and the transformation tetragonal phase of ZrO_2 to monoclinic phase led that to increase in hardness value more than AT specimen as shown in table.3.

Table 3: Microhardness before and after Erosive Oxidation Tests

Materials	Microhardness HV _{0.3}	
	Before Oxidation	After Oxidation
1-AT 600°C	454	610
2-AT 700°C	454	645
3-AT 800°C	454	690
4-AZ 600°C	483	723
5-AZ 700°C	483	765
6-AZ 800°C	483	812

Thermal Grown Oxide (TGO) Morphology

Examination of the samples cross-section after cyclic oxidation, but prior to final failure, reveals that the

undulations are located in the regions where the top layer is separated from the bond layer. As a result of applying more Al_2O_3 in the coating ($\text{Al}_2\text{O}_3+5\%\text{TiO}_2$) as top coat, also low porosity, a uniform, dense and thin TGO (dark zones) layer can be obtained in comparison with $\text{Al}_2\text{O}_3+15\%\text{YSZ}$. This can be a reason for the lower diffusion of oxygen into the TGO layer at high oxidation temperature as the detrimental oxides cannot be extensively formed within the bond coat.

The thicker TGO layer incorporates higher stresses. Hence, the crack formation at the interface of TGO/bond coat. It can be said that the oxygen can penetrate into the micro-cracks and some porosity towards the 4NiCr5Al layer during the thermal cycling and then the internal oxidation of 4NiCr5Al layer can occur as shown in Figure 6, the internal oxidation of the 4NiCr5Al layer with thicker TGO is much higher than thinner TGO.

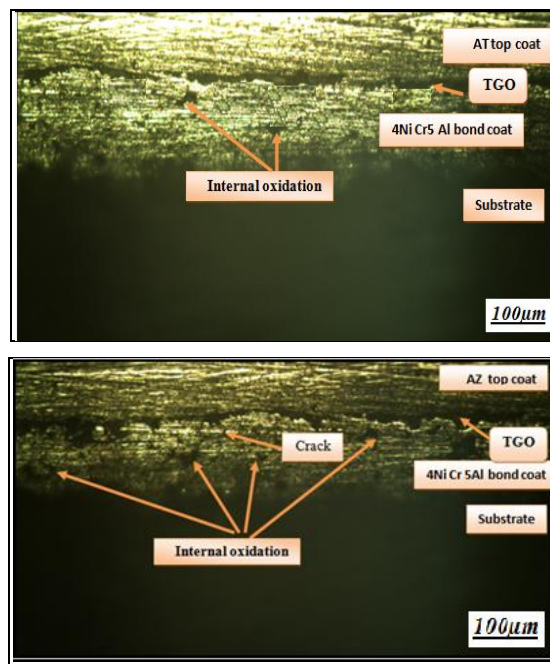


Figure 6: Illustrate Cross-Section Microstructure of the TBC - Coated Sample after 15 H at 800°C Showing TGO Morphology

CONCLUSIONS

Two types of ceramic composite coating were prepared by using mixtures of $\text{Al}_2\text{O}_3+5\%\text{TiO}_2$ and the other type $\text{Al}_2\text{O}_3+15\%$ (7-8YSZ). The coatings were HVOF-sprayed onto Monel 400 substrate pre-sprayed with 4NiCr5Al layer. The system studied and the following conclusions were made:

- Porosity and hardness of $\text{Al}_2\text{O}_3+15\%$ (7-8YSZ) is more than $\text{Al}_2\text{O}_3+5\%\text{TiO}_2$
- Adherence index of $\text{Al}_2\text{O}_3+5\%\text{TiO}_2$ is more than $\text{Al}_2\text{O}_3+15\%$ (7-8YSZ).

The specimens subjected to high temperature oxidation were studied at (600, 700, 800) °C for 15 h by XRD and OM and the following results were achieved:

- $\text{Al}_2\text{O}_3+5\%\text{TiO}_2$ has higher oxidation resistance than $\text{Al}_2\text{O}_3+15\%$ (7-8YSZ)
- Phase transformation is accompanied by volume expansion, leading to compressive stress during oxidation. It is mentionable that the compressive stresses lead to increase of the hardness.
- The study reveals that the $\text{Al}_2\text{O}_3+15\%$ (7-8YSZ) have higher hardness than $\text{Al}_2\text{O}_3+5\%\text{TiO}_2$ after oxidation test.

- A uniform, dense and thin TGO layer was formed on bond coat when ($\text{Al}_2\text{O}_3+5\%\text{TiO}_2$) as top coat, as a result of a decrease in the amount of oxygen diffusion. Furthermore, ($\text{Al}_2\text{O}_3+5\%\text{TiO}_2$) showed better oxidation behavior
- For ($\text{Al}_2\text{O}_3+5\%\text{TiO}_2$) as top coat, the internal oxidation of the 4 NiCr5Al bond coat was lower in comparison with ($\text{Al}_2\text{O}_3+15\%\text{YSZ}$) as top coat.

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