THERMAL BARRIER COATING USING YTTRIA STABILIZED ZIRCONIA BY PLASMA SPRAY TECHNIQUE

by

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This paper deals with the tribological behavior of thermal barrier coating in engine liner and it also deals with the quality of thermally treated coating. Multilayer structure formed from top coat and bond coat which was deposited on the same type of steel substrates, in which heat treatments may improve the mechanical properties of materials both substrate and coating and enhance their hardness for many cases of ceramics through recrystallization of micro-structures. In addition, heat treatments will introduce residual stresses by different thermal contractions and lattice misfits because of microstructural changes that could lead to either premature failure or stress relaxation by new crack formations. Tribological wear properties of differently treated thermal barrier coating was examined to see the net effect of heat treatment on mechanical properties by tribometer with pin on disc mode. Indentation test at surface and cross-section of coatings are examined. The SEM and optical microscope examine several crack propagations. Energy-dispersive X-ray spectroscopyhelps to characterize thermal barrier coating. After thermally treating the sample, an examination of thermal barrier coating the oxide content was increased, sample became convex due to residual compressive stress, roughness reduces, adhesion between topcoat, and bond coat get reduced eventually. In addition to that, the surface gets cracks due to the reduction in hardness from 330 HV to 212 HV.

Keywords*: tribological behavior, coatings, thermal barrier coating, testing, microstructural analysis.*

Introduction

At present, engineering products need different coating to protect from wear, corrosion, erosion, and to improve thermal behavior. Among various surface coatings, the most challenging one is the thermal barrier coatings (TBC) [1]. It asserts that due to their high de-

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manding application areas, TBC differ from other surface coatings, in which many coatings have the aim of protecting the surface or subsurface. However, TBC are quite important for the complete integrity of engineering material. Since they operate at a very high temperature range with severe stress conditions, they have to be a good thermal insulator to prevent overheating of the component, leading to creep or thermal fatigue failure. This thermal insulating property makes TBC the perfect material to use at aircraft, automotive, and industrial engines. Improving the durability of TBC will provide many beneficial outcomes. One of them will be the coating's reliability; additionally; the efficiency of the operating component will also increase by working at optimum conditions. These reasons urge a better understanding of TBC attributes like: coating type, coating structure, and their characteristics at severe operating temperatures, which are the main cause of the failure. To get maximum efficiency from TBC, there is need for detailed investigation on failure characteristics and a continuous improvement. For example, coatings, which suffer from wear, will reduce efficiency for their specific applications. Even though, it may provide an acceptable thermal barrier, low mechanical properties will restrict application type as if they will not be able to operate at jet turbine due to high wear. By understanding, the wear behavior of TBC, there is a need for an understanding of premature and abrasive failure mechanism that may improve for the best performance. Based on the information the low thermal conductivity property of TBC helps to make suitable coating for hot components [2]. To achieve this goal, TBC are generally composed of complex multilayer structure, at the bottom of the TBC metallic bond coat is formed to provide oxidation and corrosion resistance (NiCrAlY). At the top, the ceramic layer of yttria-stabilized zirconia (YSZ) is in presence. Thermally grown oxide (TGO) will form between the top and bond coat, a reaction product. The substrate is generally a super alloy (Ni, Co-based, *etc*.) and cooled from inside to provide a thermal gradient necessary for heat transport. Concerning deposition technique, conventional atmospheric plasma spraying (APS) or electron beam physical vapor deposition (EB-PVD) are used as mentioned [3]. The APS will provide a thicker and porous coating, which is advantageous for thermal insulation with the drawback of low mechanical properties. The crack formation will occur normally to heat the flow, which offers thermal insulation but decreases thermal fatigue life [3, 4]. The EB-PVD will provide thin and less porous columnar structured coatings with better mechanical properties but its low deposition rate and high cost make their utilization very particular. Crack formation is parallel to heat flow, which decreases thermal insulating property but will improve thermal fatigue life.

Recent studies have shown that plasma spray physical vapor deposition (PS-PVD) is a new encouraging technology. It has a high deposition rate property of APS and columnar structure of EB-PVD technique [4]. This technology rests on low pressure plasma spray process but it has lower operating pressure and high power plasma gun. Due to the high working temperature, there is the utilization of grain-sized particles occurs. Plasma current is able to pass around complicated shapes of the substrate by forcing into the sheltered regions, whereby composing the PS-PVD a non-line-of-sight layer process happens. The structure of the coatings strongly relates to spraying distance. As the distance increases, the porosity also increases in the coating with more columnar and less lamellar micro-structure. This promising technique could consolidate both advantages of EB-PVD and APS method in which, high-grade thermal insulation and strain tolerance help to improve the TBC performance [3] that TBC failure occurs due to many different mechanisms. Since TBC operate at severe conditions many phenomena befall; diffusion, oxidation, elastic deformations, creep, thermal fatigue and fracture, thermal conduction, phase transitions, and sintering. According to these phenomenon's failure of TBC occur due to crack propagations, stresses at the topcoat, possible spallation at bond coat substrate interface, thickening of TGO (bond coat/TGO and topcoat/TGO interfaces) and cracks in the bond coat. In addition, sintering of top coat due to high temperature may decrease its strain tolerance by reducing porosity. From those mechanisms; besides residual stresses, one of the main concerns for the failure of the TBC is the TGO growth and interactions with interfaces. In relation to improve lifetime and prevent premature failure of the TBC, there is a need for deduction of more erudition about TGO behavior. In general, the bond coat is a layer deposited through plasma spray or EB-PVD with a thickness range of 70-150 µm in more than only one form. It consists of Ni, Cr, Al, Y, Pt, and Co. Even though the bond coat is repellent to oxidation, the high operating temperature range of TBC and porous coating of the ceramic top coat helps infiltrate oxygen. An oxide layer is available, called TGO, with a thickness of 1 to 10 µm. It helps reduce TGO formation and makes a forcing bond coat to produce $\propto$-Al_2O_3 to form a diffusion barrier and prevent more oxidation, making TGO more adherent [3]. The growth of TGO is parabolic until failure occurs with the columnar zone near the bond coat and near the equiaxed topcoat [4]. Inward diffusion of oxygen will lead to the growth of TGO through the bond coat, and outward diffusion of Al result in a new layer of TGO next to the top coat. The stresses that are a presence in TGO are the key elements for premature failure of TBC. Residual stresses due to thermal contraction and the growth of TGO will be the main sources of stresses, which distributed over imperfections. Thermal treatments modify those residual stresses [3]. Strain energy due to residual stresses at the TGO layer will show a linear trend with TGO thickness and quadratic trend with TGO stresses. As the thickness of the TGO grows, tensile stress induces to bond coat TGO interface and lead to cracking at the interface. Also during thermal cycle roughening of interfaces will occur, which lead to implantation of planes. Even though, the thermal residual stress at top coat due to contraction is at the compressive form it could not compete with tensile inducing residual stresses of TGO due to strain tolerant impurities at the top coat. Estimating mechanical attributes of TBC can accomplish with many tests. Wear test will provide information about the tribological properties of TBC by evaluating the material loss due to abrasion. Scratch test will give crack propagation at the cross-section of the coating by practicing scratches. Pull on analysis helps to appraise adhesion between the coating and the substrate where there was the binding of top and bottom and pulled until failure. Indentation test executes to find the hardness of the coating. In this test, there is an increased progression of load, which helps to counterbalance elastic recovery and extension of the deformed zone, which cause a misleading outcome. By the amount of load and indentation time, cracks may form at the surface of TBC, which relates to instantaneous HV drops and stress leisure .Wear properties of coatings that relate to the hardness of the surface, however, premature failure mechanism is also caused huge importance for wear resistance. The wear test helps to assess the effect of thermal treatments in the topcoat micro-structure, in which there is an introduction of assumed crystallinity available. In addition, spallation, due to TGO growth and cracks because of thermal residual stresses could lead to unanticipated failure of TBC, which shall be pondered to wear test. Furthermore, induced thermal cycles could regulate impurities at the top coat and reduce strain tolerance of the top coat in which load carrying capacity of the top coat is reduced [5, 6].

In order to investigate the effect of heat treatment on the tribological properties of TBC, the following experiments were available and arranged. Energy-dispersive X-ray spectroscopy (EDS) helps to achieve characterization of TBC to derive composition knowledge of layers. Then there will be a performance of pin of disc and indentation test available and SEM will conclude the assessment. Later there will be a comparison in results and it acquires the determinations about the effect of heat treatment over TBC materials.

Experimental detail and procedure

The application of wear and indentation tests on top of the coatings, before performing the tests, compositions at the top of the TBC are considered through EDS, the content table is formed as a complementary to composition information. Through gathering Kaplot from EDS analysis curves, a composition chart for the top of the coatings could be prepared, as shown in tab. 1. The equation of yttria based zirconia is $(ZrO_2)_{1-x}(Y_2O_3)_x$. As shown in figs. 1(a). and 1(b), it is possible to observe the physical differences.

Table 1. The EDS results of the specimen

Coating type	Weight O [%]	Weight Y [%]	Weight Zr [%]
As coated	20.60	5.82	73.58
Thermally treated	26.43	0	73.57

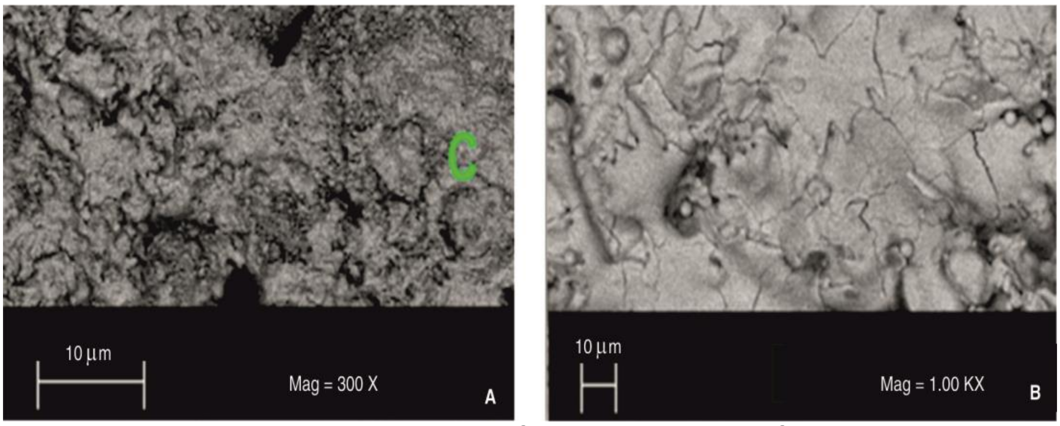

Figure 1. Thermally treated surface (a) and as coated surface (b)

Sample description

The sample are coated with APS technique by the topcoat composition of 26 w.% Oxygen and 74 w.% Zirconium. Exposed to 20 thermal cycles before obtaining the sample, and then applied one more cycle at 800 °C for 5 minutes in the oven from Carbolite. Then, it quenches to room temperature with deionized water to prevent any unwanted ionic depositions on the specimen.

As coated sample

Coated with APS technique by topcoat composition of 20 wt.% of Oxygen, 6 wt.% of Yttrium, and 74 wt.% of Zirconium.

No thermal treatment applied and it works in undeviating conditions in this experiment. White specimen, fig. 2(a), is as coated. It is seen that thermally treated specimen has a bending curvature due to compressive residual stress, which results from thermal treatment where contractions occurred due to different expansion coefficients is shown in fig. 2. The color difference relates to different interactions of light with different micro-structures of coatings due to the heat treated sample's thermal treatment process in which the crystallization phenomenon occurs.

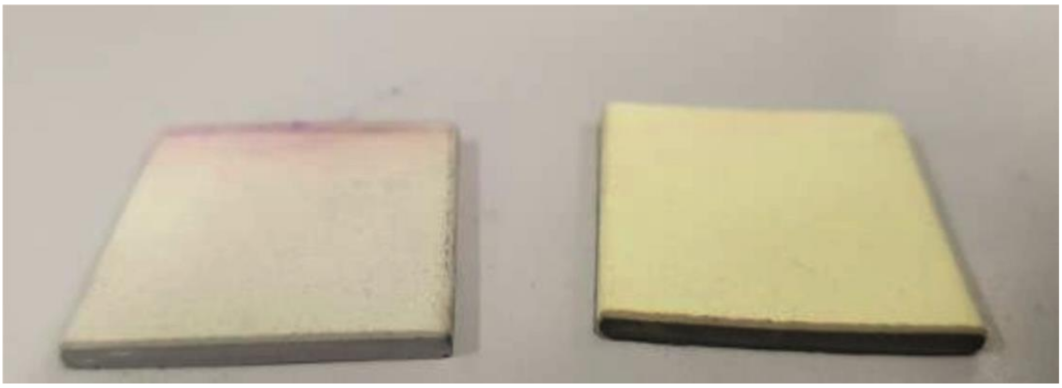

Figure 2. Specimen (a) as coated and (b) is thermally treated

Thermally treated sample

Coated with APS technique by the topcoat composition of 26 wt.% Oxygen and 74 wt.% Zirconium. Exposed to 20 thermal cycles before obtained the sample, then applied one more cycle at 800 °C for 5 minutes in the oven from carbolite. It is then quenched to room temperature with deionized water to prevent unwanted ionic depositions on the specimen.

Wear test

Wear test was done by using Tribometer by CSM with the ball on disc mode. The material of the ball was Al_2O_3 with a diameter of 6 mm. With this technique, abrasive wear response between coating and ball examined. The friction coefficient was evaluated via calculating resistive force, which is the ratio between friction forces over tangential force. The instrument does the calculation. Tangential force is the force perpendicular to the contact normal, which is in the opposite direction to sliding direction.

The tribometer's operation began with the experiment being placed in the specimen holder. Height of the pivot arm is set by placing the arm at equilibrium. The equilibrium state of the pivot arm is tested by making a small tapping against the arm and observing its movement. This will provide that the weight on the material is the load that is desired. For Specimens 1 and 2, exactly 1 N load is applied which produces friction, thus leads to wear. To ensure the force, a rubber ring is placed on the ball holder. Additionally, that rubber ring prevents the noise. An excessively applied force will absurdly stop the experiment due to the excess amount of tangential force. On the contrary, increasing the linear speed will decrease the tangential force.

On the first trial with the as coated specimen, after the first 15 minutes, the experiment failed due to high tangential force. High tangential force is dangerous for our instrument, thus, the program stops the experiment in order to prevent overheating. With some investigation, the reason for the failure is determined that the linear speed is low. This could be solved by increasing the linear speed depending on the wear mode, which is an unknown [7]. After adjusting the linear speed from 0.2 m/s to 0.25 m/s. As linear speed increase, probability to mechanical interlock decreases [7]. To avoid overlapping on the track of the preceding run and obtain new reliable results, the radius of the path increased from 3.49-4.5 mm manually to get results from a new area. By increasing the track radius, the total number of laps is also increased. As a result, heat due to friction between ball and coating distributed over a larger circumference, which reduced the overheating. Unfortunately, increasing only the linear speed was not enough to reduce tangential force. To solve this problem, on the second trial of the wear test, the applied load decreased from 2 N to 1 N. Totally two modifications are made in order to measure wear and the same condition tried to apply to the thermally treated specimen tab. 2. The main problem, the thermally treated sample was not flat like as the as-coated specimen that curvature was caused by thermal stress. On the coating, there is compression and at the substrate, there is tension. As a result, wear test for the thermally treated sample was not successful since there was a high tangential force IntRunX software used to operate CSM Tribometer.

Table 2. Wear test specification

	Load [N]	Radius [mm]	Distance [m]	Linear speed [ms^{-1}]	Acquisition rate [Hz]
Thermally treated	1	4.5	343	0.25	10
As coated	1	4.5	1500	0.25	10

Results and Discussions

Micro-structure Analysis

An optic microscopy, fig. 3, helps to make the micro-structure of the as-coated and thermally treated samples. Both samples are polished and then etched. In the as-coated images, it is possible to observe the difference between interphase and the centre of the sample. Unlike the as-coated sample, the crystallization process reorganizes units by the temperature that provides the necessary energy to mobilize unit cells. As a result, there is the formation of ordered structure, grain boundaries, and those are observable by optical microscope for thermally treated coating, which could increase the toughness of ZrO_2 [8].

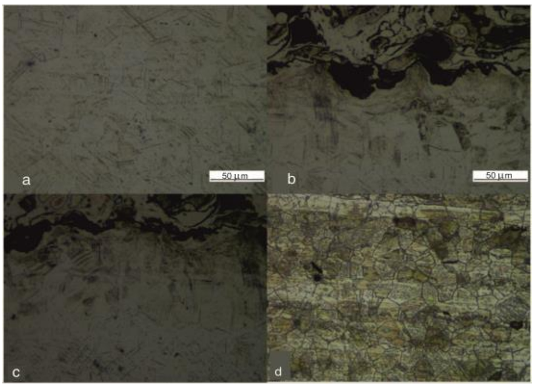

Figure 3. (a)-(c) As coated micro-structure and (d) as thermally treated micro-structure

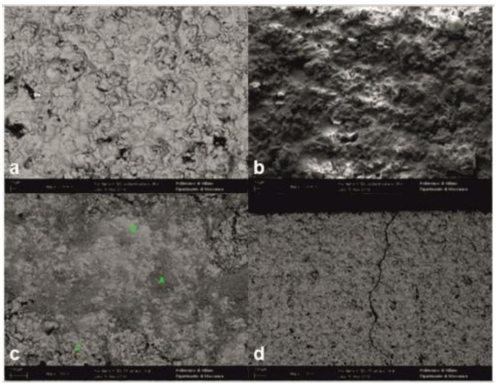

Figure 4. (a) and (b) the SEM image of built sample surface morphology and (c) and (d) as thermally treated sample surface morphology

Surface morphology analysis

From the above SEM image, fig. 4, it is clear that the surface is porous before thermal treatment. However, there were no cracks. However, after the thermal treatment, it is evident that from the fig. 4(d) image that cracks start to propagate, especially from the corners, due to compressive residual stresses, which becomes the cause for the thermal contractions after heat treatment. We also found a different texture at different locations, fig. 4(c). On EDS analysis, tab. 3, from all the three locations, there is an observation available that there were minor variations in the composition. At spots A and C, fig. 4(c), no traces of Yttrium was detected and at spot B, there was a reduction in Zirconium content but an increase in the oxide content.

Table 3. The EDS analysis of the thermally treated sample at three different locations

Location	Weight O [%]	Weight Al [%]	Weight Y [%]	Weight Zr [%]	Total
A	26.23	--	--	73.77	100
B	30.08	0.45	5.32	64.14	100
C	26.43	--	--	73.57	100

Surface roughness analysis

From the figs. 5(a) and 5(b) red points showing the peak points and blue ones are the surfaces' pit points. In which, it can clearly be seen that as coated one has more asperities. Thermally treated one has more intense pit and peaks.

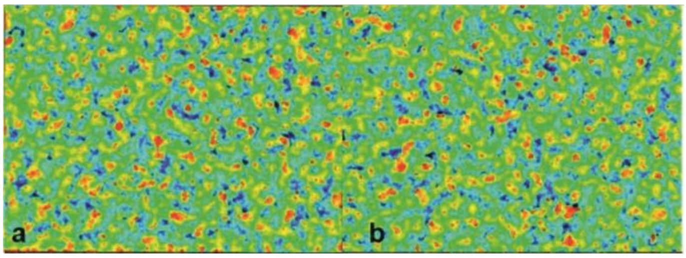

Figure 5. (a) As coated and (b) as thermally treated
(*for colour image see journal web site*)

From this tab. 3, it can be resolved that as coated TBC has a high average roughness due to high S_a value compared to thermally treated coating. The S_a indicates arithmetical mean high of the surface asperities, and S_z stands for maximum height. However, even though the as-coated sample has a higher roughness, the thermally treated coating has a higher maximum height between pits and peaks [9, 10].

Table 3. Arithmetical mean high of the surface asperities, S_a, and maximum height, S_z, of as coated and thermally treated sample

	S_a [μm]	S_z [μm]
As coated	8.857	97.608
Thermally treated	8.759	124.281

Wear test analysis

The tribology test helps to assess the surface status of specimens by mean of the friction coefficient. The wear test specimen is shown in fig. 6. The average value of the as-coated sample's friction coefficient is 0.482, much higher than the result obtained for thermally treated sample 0.253. At the beginning of the test, the contact occurs at only a few points called asperities. These points deform elastically where they touch, which results in very high initial values of friction coefficient [7, 11]. However, as the samples become the real effort for the real contact area between the ball and the coating surfaces increases and the friction coefficients rise gradually.

From figs. 7(a)-7(c) wear trends, it can be said that the friction coefficient of the as-coated sample is reduced because of the thermal treatment. This can be related to the decrease in the average value of roughness, S_a. As the asperities at the surface of the coating are diminished, the ability to have a mechanical interlock, which increases the adherence between two surfaces with the contact surface is also decreased. This decrease in mechanical interlocking can make relative movement easier. Therefore, in this case, the abrasive mechanism determines the friction coefficient. The thermal treatment due to the lowering of roughness becomes the reason for the increase of the contact area between two surfaces [12]. Thus, the friction coefficient slows down. Therefore, it is true that adhesive forces are not dominating in this situation. This becomes the reason for the presence of low surface energies at the contact surfaces.

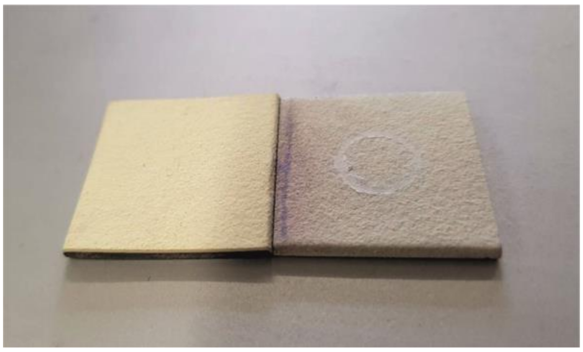

Figure 6. As coated specimen after wear test (right) by tribometer and thermally treated specimen (left) before weartesting

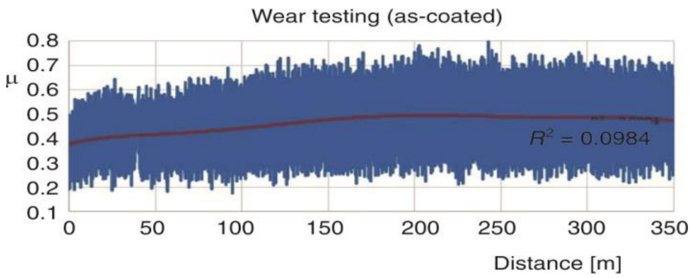

Figure 7(a). Wear test of as-coated sample up-to 350 m, friction coefficient with Respect to distance

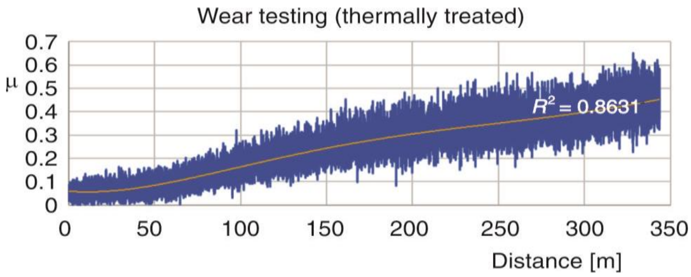

Figure 7(b). Wear test of thermally treated, friction coefficient with respect to distance

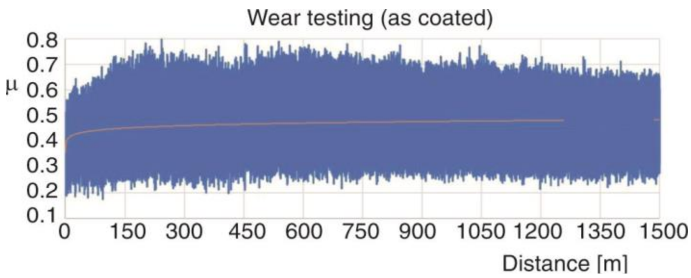

Figure 7(c). Wear test of the as coated sample upto1500 m, friction coefficient with respect to distance

From the fig. 8, we could even differentiate the wear test path on the sample during SEM analysis. It has a smooth sliding effect over the surface. The EDS analysis helps to confirm the effect. We could find the traces of aluminum 1.38% by weight on these locations. This Al may be the material loss from the ball tip of the wear testing apparatus that is in contact with the specimen during the test.

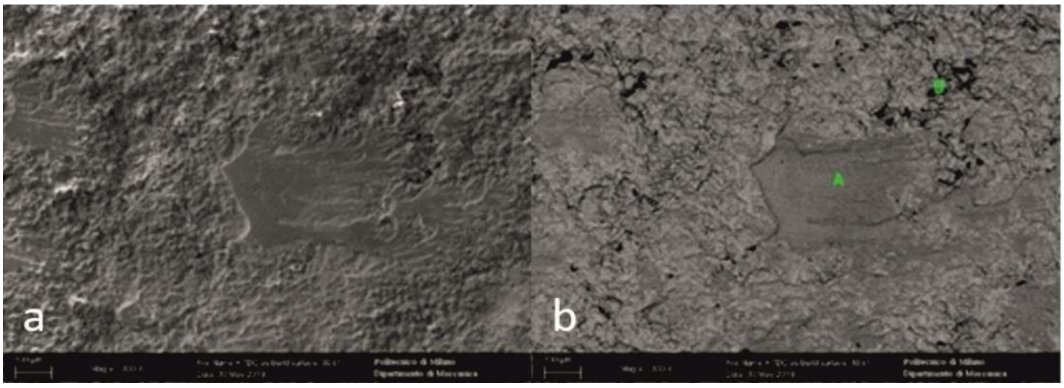

Figure 8. The SEM analysis of the wear test

Hardness test analysis

The results of the hardness test are available in the tab. 4.

Table 4. Hardness results of specimens

Measurement	As coated	Thermally treated
1	330	212
2	393	332
3	438	373

From fig. 9, after being thermally treated, the layer's hardness value slightly decreases compared to the as coated sample. It says that the thermal treatment process does not significantly affect the hardness of TBC, which is in agreement with the result from [1]. The hardness is recorded using Vickers hardness testing machine [12].

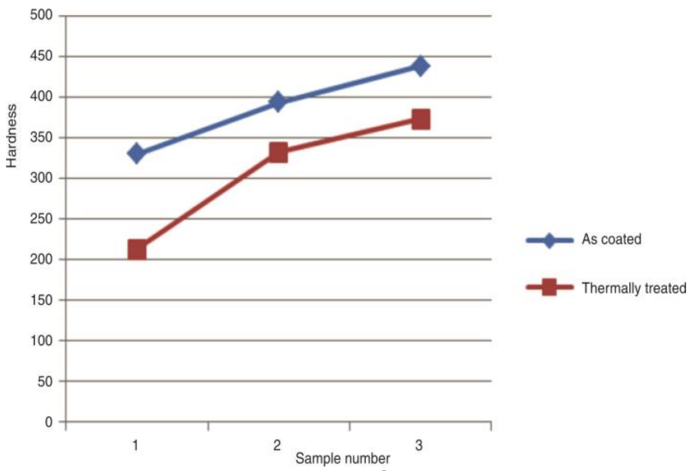

Figure 9. Plots of hardness

The abnormal values 438 HV and 212 HV do not take into account, as they do not represent the hardness of the TBC. Cracks are propagating from the indentation mark for the thermally treated sample where the hardness value of 438 HV is available. The indenter breaks the top coat layer that it is measuring the hardness of the material, then the operation goes on and non-correct value of hardness is available. The lowest value 212 HV for the as-coated sample is also available at the imprint where coating starts to fail. The consideration that a plasma spraying method helps to deposit the TBC tested in this laboratory provides micro cracks and high porosity inside the coating.

Indentation test analysis

Figures 10 and 11 shows the indentations that help test the coating's hardness before it was thermally treated. It is notable that there was no development of cracks or tears during the indentation rather we could see the squeezed material out of the side during indentation.

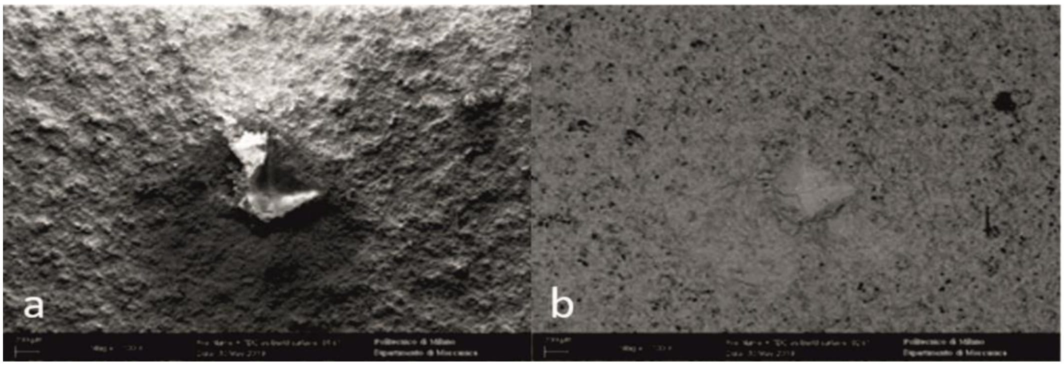

Figure 10. Indentations on the as coated sample after a hardness inspection

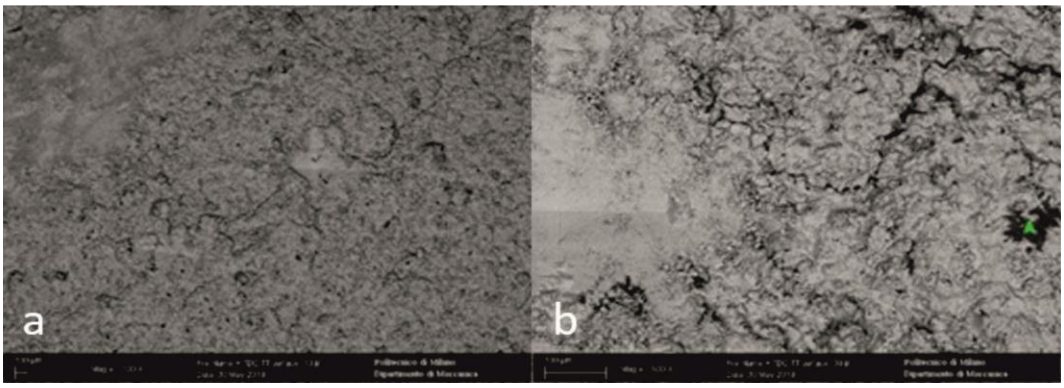

Figure 11. Conduction of Indentation hardness test on thermally treated sample

Hardness test was carried out after the samples were thermally treated and it was found that the coating has become more brittle. On close observation, we found that cracks originated from the point of indentation and started to propagate in either of the direction. These cracks occurred due to stress relaxation at the contact area during the indentation test. At those points, applied stresses are higher because of the edge effect and lead to the release of residual compressive stresses by forming cracks.

Some black spot appeared after thermal treatment and after EDS analysis at the spot A, it was found that it was just contamination and there were no changes in composition at the particular site.

Conclusion

From the aforementioned experimental analysis, the characteristic properties of thermal barrier coating on the substrate are thermally treated and as built. The comparison studies between them can differentiate the various behavior changes such as micro-structure hardness, roughness, toughness, wear, surface morphology, and compositional changes of the coating in two different conditions are discussed as follows.

- The compound thermally treated made some changes in the chemical composition as well as its physical appearance. Amount of oxide increases due to TGO growth; coating got a convex curvature due to residual compressive stress. In addition, due to thermal treatment crystallization occurred at the surface, which leads to opaque appearance.
- While comparing the thermally treated roughness and as a coated specimen, thermally treated has less roughness. The average surface asperities are reduces from 8.857-8.759 μm. Because of this, it expects to have a lower friction coefficient due to a smoother surface. From wear test data, it seems that the thermally treated sample has a higher friction coefficient. The reason is also visible on fig. 10, which is the prevention of mechanical interlocking.
- The SEM images of the cross-sections of the TBC show that after the heat treatment process amount of oxide is increased because of TGO. Since an oxide layer decreases the layer's surface energy, adhesion between the top coat and bond coat will be lower. Because of this, any applied stresses thermally or mechanically will trigger the spallation of the coating.
- Thermal treatment cycles induced residual stresses to the coating due to thermal contraction between substrate and coating. Since the coating's curvature is in convex shape Figure 9 these residual stresses are in compressive form. In addition, from fig. 5 it can be in observation that cracking occurred due to compressive stresses. Due to the way of the deflection, there is compression on the coating and tension on the substrate. Additionally, from indentation test, it was in observation that relaxation of these compressive stresses was done by crack formation at the surface, which reduces the coating's hardness due to early failure of the coating by crack formation.
- The thermal cycle of APS coated TBC can suffer from spallation due to compressive stresses, with additional probability of premature failure as a result of TGO growth. Also, the friction coefficient of the coating is decreased by applying thermal treatment.

Further improvements can happen to increase the properties of TBC.

- In this experiment, the key elements, which are dangerous for TBC, are compressive residual stresses at the top coat and oxidation between the interlayer of the coatings. The prevention of oxidation occurs at the interfaces by having a less porous structure that prevents the permeation of oxygen into the coating. There is an achievement is possible by forming a coat with a PVD technique. In which coating will have a columnar and less porous structure compared to APS technique coating.

Nomenclature

APS – atmospheric plasma spraying
EDS – energy dispersive X-ray spectroscopy

EB-PVD – electron beam physical vapor deposition
PS-PVD – plasma spray physical vapor deposition
TGO – thermally grown oxide
TBC – thermal barrier coating
YSZ – yttria stabilized zirconia

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