IMEKO TC15 - Experimental Mechanics - Proceedings of the
11th Youth Symposium on Experimental Solid Mechanics
Brasov, ROMANIA, 2012, May 30-June 2

COMPARATIVE RESEARCH ON THE STRUCTURE AND PROPERTIES OF ZrO_2/20% Y_2O_3 LAYERS OBTAINED WITH PLASMA SPRAY DEPOSITION METHOD

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Abstract: *Ceramics and ceramic materials are materials with promising technical applications and with relatively high characteristics in terms of mechanical strength, but at the same time very low properties due to the fragility and the cracks appearing in the cooling layer.*
The cracking resistance is important not only for the technical products which operates in the mechanical thermal loads domain but also for structural components, cracks of which fragility is unacceptable even in arbitrary tasks.In this paper its being revealed a new concept of TBC with an exfoliation prevention of the sprayed layer and it consists in an adherent layer of NiMoAl (90-5-5) sprayed through an electric arc and a top coat of ZrO_2/20%Y_2O_3*, deposited by atmospheric plasma spraying (APS) on specimens of Ni base superalloy, of which the aircraft turbine blades are made. There were made some attempts of spraying at different distances, namely: 100 mm respectively 110 mm. These samples had been then subjected to mechanical bending tests to observe adhesion to the substrate. Highlighting and interpretation of structural changes caused by the tests carried out have been made using modern methods of structural analysis*

Key words: ZrO_2/20% Y_2O_2 *, SEM, ANSYS.*

1. Introduction

Plasma spray represents one of the most important achievements in surface engineering, domain in which a big progress was noted in the last few years. [1]

This process is in principle based on spraying molten or semi-molten materials on a substrate to achieve a covering layer. Powdered materials are injected into a very high temperature plasma jet; they are immediately warmed and deposited with a very high energy. Materials at high temperatures are hitting the substrate surface and rapidly cooled to form a covering layer. This process, correctly developed, is also called "cold process" because the substrate temperature can be kept low during the process, thus avoiding

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destruction, structural transformation or distortion of the substrate material. Since the turbine blades need strength to function in a high temperature regime, thermal spraying is a optimal solution for getting a good resistance to wear, corrosion, thermal shock and avoiding premature exfoliation.[2]

This paper presents a new concept of thermal barrier coating for the prevention of delaminating of the sprayed layer. This consists in an adherent layer of NiMoAl (90-5-5) sprayed with electric arc and a top coat of $ZrO_2/20\%Y_2O_3$ (purity of 80% Zr,), deposited by atmospheric plasma spraying (APS) on samples of Ni base superalloy, of which the aircraft turbine blades are made.

For the spraying of the samples there were used two different distances namely: 100 mm respectively 110 mm. These samples were then subjected to mechanical bending tests to observe adhesion to the substrate.

2. Experimental procedure

The thermal barrier coating was obtained using atmospheric plasma spraying deposition (APS). The ceramic powder sprayed on the samples is zirconium (stabilized with 20wt% yttrium) using a Sulzer Metco SPRAYWIZARD-9MCE installation. The bond coating was sprayed with an NiMoAl powder on rectangular samples by electric arc method using an Sulzer Metco Smart Arc 350 device, on samples of Ni super alloy, cleaned in an ultrasonic bath with acetone and sand blasted with electro corundum. The size used for the samples was 8x30x2 mm.

To highlight the results, analysis were performed using electron microscopy with the QUANTA 200 3D DUAL BEAM electron microscope. The CAD models for the samples and the ceramic layers on both spraying distances and the assembly between the sample and the ceramic layer were made using CATIA V5 R19 program. The finite element analysis was performed using the static structural module from the ANSYS 13 program.

Deposition parameters for atmospheric plasma spray (APS) are presented in Table 1, and intermediate layer parameters with Ni Mo Al deposited by arc are shown in Table 2.

Technical parameters Table 1

APS	100mm	120mm
Cooling water debit	8,7 bar	8,7 bar
Velocity of rotation	55 rot/min	55 rot/min
Electrode voltage (U)	60 V	60 V
The intensity of the gas Plasma (A)	600 A	600 A
Composition of plasma	46,1%Ar/ 13,51%H_2	46,1%Ar/ 13,51%H_2
Spraying distance	100 mm	120 mm

Sulzer Metco Smart Arc 350 Table 2

U	31V
I	200A
Air pressure	60 si

3. Experimental results

To perform the bending test a three point stand was used. The sample was caught precisely between the support rollers and centrally positioned with the bearing axis. This was necessary so no slides would appear during the test. The loading speed was constant and equal to 0.5 mm/min. [3] The dynamic load and flexibility transducers were calibrated with a coordinates potentiometer. The three-point bending test can be used on small samples. The test was undertaken until the sample presented a macroscopic crack in the ceramic layer.

Fig. 1. *Sample in working.*

The maximum tension was calculated using Navier's relation, in which the normal maximum tension is equal to the ratio between the maximum bending moment and the axial resistance modulus, using the following equation:

$$\sigma_{max} = \frac{M_{i_{max}}}{W_z} \qquad (1)$$

The electron microscope was used to record the evolution of deformation and cracks in the layer after the test.

3.1. Micro structural characteristics before and after being subjected to bending

A sprayed coating partially stabilized with 20% Y_2O_3 has a porous and lamellar structure and consists of splats perpendicular to the surface. This porous structure allows the increasing of the thermal insulation and the cracks allow a better adaptation of the material to the tensions that arise. The adhesion of the layer reduces the thermal expansion discrepancies between the coating and substrate. This leads to an increased lifetime for the component.

The presence of cracks also increases the tolerance to deformation and improves thermal shock resistance for TBC's.

After the air recovery, all coatings show a sintered structure; this leads to a reduction in porosity. This is in agreement with other studies. [3] In the Figures 2a/2b and 3a/3b are presented SEM images in cross-sectional of the layers for both spray distances.

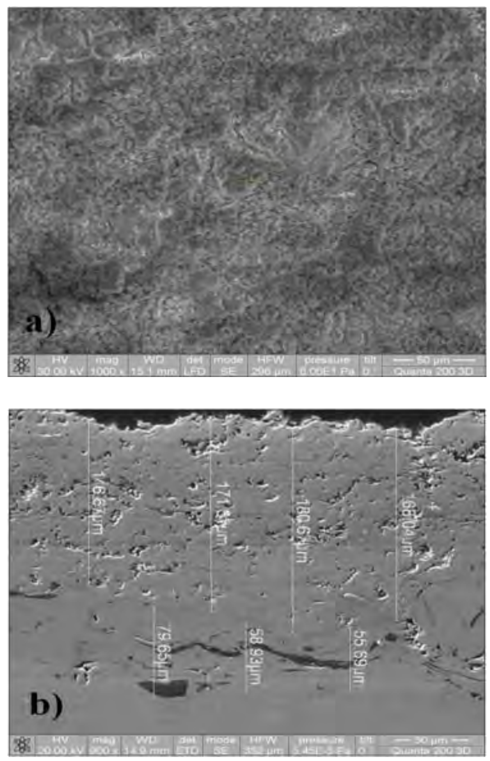

Fig. 2. *SEM* images *for the layer obtained at the 100 mm spraying distance a) surface analysis and b) throughout cross section*

The effect of sintering will determine the increased thermal conductivity, the elasticity modulus and a decrease in tolerated stress (Figures 2a and 3a).

It can be seen that the coating has a porous and lamellar structure (Figures 2b and 3b). In addition the deposited layer has a good compactness and adherence to the addition material. The structural attack revealed that there is a diffusion zone at the interface between the deposited layer and the substrate.

The chemical composition of the microstructure is presented in the images with a gray variation and the pores appear very dark.

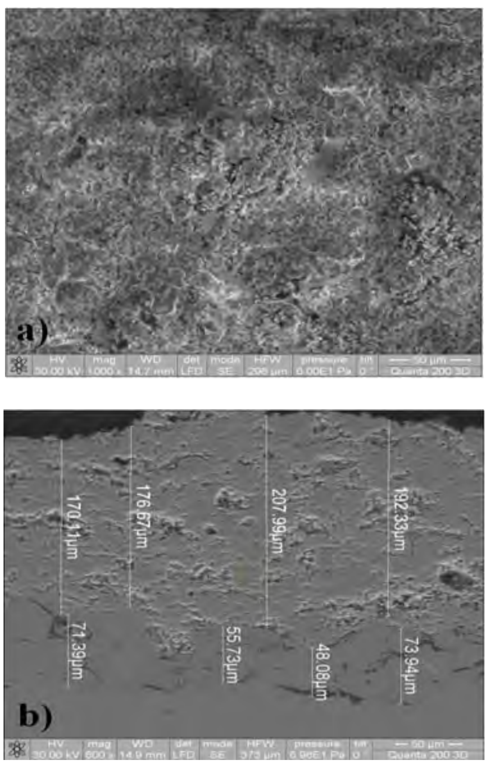

Fig. 3. *SEM images for the layer obtained at the* 120 *mm spraying distance a) surface analysis and b) throughout cross section*

3.2. Crack observations

One of the difficulties that arise in the bending test is to mount the sample in the support. Due to the fragility of the material it is difficult to avoid the initiation of a crack during this stage of the test.

In Figures 4 and 5 are presented SEM images of the surfaces at different magnification with cracks after the mechanical bending test. Cracks are perpendicular to the applied force. [4]

From the tests that were carried out on the two samples it can be seen that their behavior is different. In the case with the spraying distance of 100 mm the maximum applied force for which the material has withstood is of 1939.71 N. For the sample with the spraying distance of the 120 mm the maximum applied force for which the material withstood is of 2163.72 N. It can be concluded that for the spraying distance of 120 mm the analyzed material withstands a higher applied force.

In Figures 6 and 7 there are presented SEM images of the material from the edge of the sample to see if exfoliation in the substrate layer appeared after the mechanical bending test. In the figure 6.a. can be observed a visible crack, but it has not reached the base material only the interlayer. This fact can be seen from the EDAX analysis of the crack. (Figure 6 a/b)

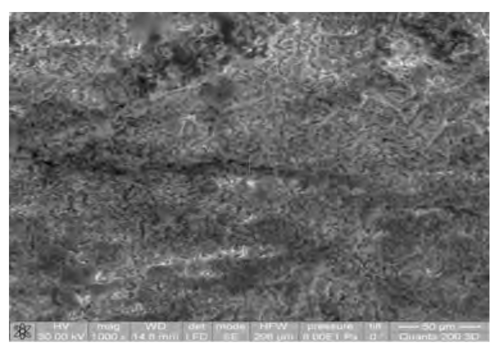

Fig. 4. *SEM* image *of the surface layer with cracks for the 100 mm spraying distance*

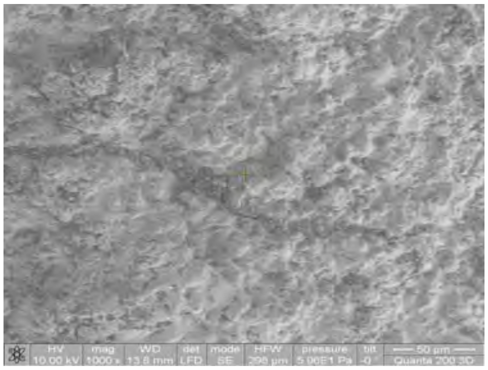

Fig.5. *SEM image of the surface layer with cracks for the 120 mm spraying distance*

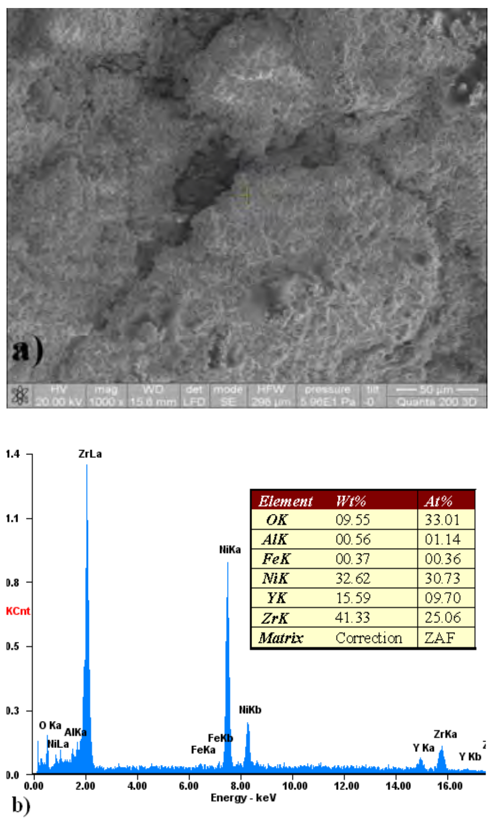

Fig.6. *SEM image and EDS analysis of a crack on the edge of the sample in Figure 6.a., with the spraying distance of 100 mm*

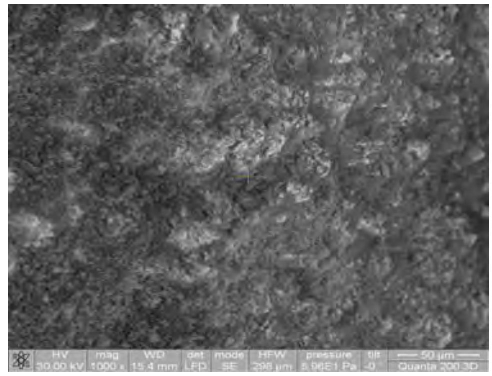

Fig.7. *The appearance of the crack on the edge of the sample produced by bending, for the 120 mm spraying distance*

In Figure 7 it is observed that layer was not exfoliated and lasted up to a value for the force of 2163.72 N.

To determine the tension that appears in the material a analytical calculus was made, with the results shown in Table 3 for both spraying distances (Figures 8.a/b.). To achieve the FEM analysis was used the ANSYS 13 program. The CAD model of the assembly was imported into ANSYS from CATIA. For the mesh network were used tetrahedral elements. The mesh network is composed of 16,433 elements and 40,675 nodes. The results for both spray distances are shown in Figure 8a and 8b.

Table 3

Plasma spraying distances	100 mm	120 mm
Elongation[mm]	0.877	0.714
Force [N]	1939.71	2163.72
Iz [mm^4]	5.33	5.33
K	0.256	0.256
$\sigma_{x\ max}$ [MPa]	496.56	553.91
E [MPa]	38109	52214.89

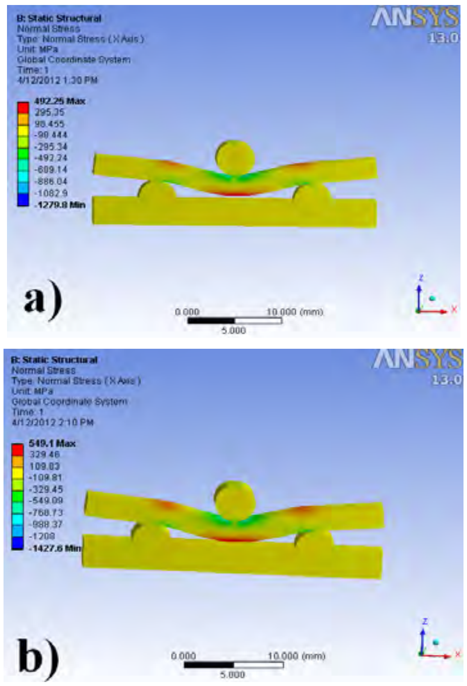

Fig.8. *Tension distribution for the sample: a) with the spraying distance of 100 mm and b) with the spraying distance of 120 mm*

4. Conclusions

Taking into account the characteristic spraying parameters of the deposition installation and stresses for which the deposited layers will have to withstand the following working parameters were established and presented in Table 1 and 2 for both spraying distances.

Using scanning analysis with electron microscopy it was observed that the coating sprayed at the distance of 120 mm is thicker than the one sprayed at the distance of 100 mm and has a better adhesion to the substrate.

In the case of the spraying distance of 100 mm the maximum applied force for which the material withstoods is of 1939.71 N and the maximum normal stress that appears is of 496.56 MPa.

In the case of the sample with the spraying distance of 120 mm the maximum applied force for which the material withstoods is of 2163.72 N with a maximum normal stress of 553.91 MPa.

The analytical calculus has similar values as the results presented in Table 3.

Acknowledgements

We thank: Karlsruhe Institute of Technology (KIT) Research Center, Institute for Pulsed Power and Microwave Technology (IHM) Mr. dr. Adrian Jianu.

We thank SC AEROSTAR SA from Bacau for providing the super alloy substrates.

We acknowledge that this paper was realized with the support of EURODOC "Doctoral Scholarship for Research Performance at European level" project financed by the European Social Found and Rumanian Government.

References

1. P. Motoiu, *Tehnologii şi pulberi pentru obţinerea straturilor metalizate termic,*
2. N. Curry, N. Markocsan, Xin-Hai Li *Next Generation Thermal Barrier Coatingsfor the Gas Turbine Industry*, Aure´lienTricoire, and Mitch Dorfman (Submitted May 10, 2010; in revised form September 20, 2010).
3. J.C. van den Eynden, Technische Universiteit Eindhoven, Department of Mechanical Engineering, *Microstructural Characterization of Refractory Ceramic,* March 21, 2005 - May 27, 2005.
4. J. T. PRATER AND R. W. MOSS Pacific Northwest Laboratory, Richland , *Effect of the coating structure on the adherence of sputter-deposited oxide coatings*, WA 99352 (U.S.A.) (Received March 24, 1983; accepted April 19, 1983.