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BEHAVIOR TO HIGH SPEEDS HEATING-COOLING OF CERAMIC MULTILAYER MULTIFUNCTIONAL STRUCTURES BASED ON THE ZIRCONIE PARTIAL STABILIZED WITH YTTRIA

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Abstract: *Advanced materials intended for application in aerospace, power, metallurgical industries impose composite functional materials and new methods and installations to test them. Thermal barrier coatings (TBCs) are widely used to protect components and remain the most effective thermal insulation approach and their development was focused on partial stabilized zirconia. In this paper the used material is zirconia partial stabilized with 20% yttria in place of the classic TBC stabilized with 7-8%. In order to evaluate the behavioral assessment of TB protection, attesting installation was designed and realized by the INCAS, for cooling-heating speeds up to 100^0C/s, installation included in the experimental program. In the view of the evidence regarding interfacial structural changes induced by thermal shock due to high thermal gradients the results of SEM-EDS electronic microscopy investigations were presented.*
Keywords: *SEM, ZrO_2/20% Y_2O_3, thermal shock*

1. INTRODUCTION

Gas turbine aircraft engines are operating under severe thermal, mechanical and chemical conditions. The turbine technology depends on the development of suitable materials that can operate in extreme working environments. Of all the factors acting simultaneously and causing wear, the most important is thermal shock. In the case of the hot parts which compose the turbines, the temperature varies depending on operating conditions: takeoff, landing, cruising speed, landing failure, engine shutdown during flight, etc. The use of thermal barrier coatings allows the increase of the operating temperature without increasing the temperature of the base material and the reducing of the amount of air necessary for cooling.
Taking into account the extreme operational conditions of the "hot parts" of the turbo engine it is necessary to study the material behavior at high rate speeds of heating-cooling cycles and thermal shock.

2. MATERIALS, METHODS AND INSTRUMENTATION

For experiments the following multilayer samples were used:

- Nimonic 90 support;
- NiCrAlY bonding layer (AMDRY 962) having as chemical composition Ni,Cr,Al,Y;
- ZrO_2/20%Y_2O_3TBC layer;
- The thickness of the TBC deposition is between 0,15 and 0,47 mm;
- The samples have rectangular shape with the following dimensions 2.15x30x50 mm; 2.28x30x50 mm; 2.47x30x50 mm.

The protecting layers were obtained by successive deposition. Both the bonding layer and the ceramic layer were deposited by atmospheric plasma spraying method on a 7MB type METCO installation. The spray parameters used for the deposition installation are presented in Table 1.

Table 1: Parameters of deposition

Technological parameters	NiCrAlY	YSZ (202NS)
Spray distance, (mm)	120	120
Injector	1,8	1,8
Plasma gas intensity, (A)	600	600
Arc voltage (U)	62	65
Speed of rotation (rot/min)	55	55
Argon flow (m^3/h)	50	40

In the picture below is presented the QTS2 installation, designed and built by INCAS (National Institute for Aerospace Research "Elie Carafoli") for testing materials in extreme heating-cooling cycle conditions, Figure 1.

Figure 1: QTS2-Installation for material testing in extreme thermal conditions

The Quanta 200 3D electron microscope was used to perform secondary electron images and EDS analysis, working in the Low Vacuum module at pressures ranging from 50 to 60 Pa and using the LFD (Large Field Detector) detector. The voltage used to accelerate the electron beam had the value of 30kV and a working distance varied from 12 to 15 mm.

3. THERMAL SHOCK RESISTANCE TEST

The aim of the thermal shock resistance test is to reveal micro structural changes of the tested samples. The thermal shock test is completed when macroscopic exfoliation appears and the damage is more than 25% of the TBC surface [1]. The thermal cycling has been performed at 1200ºC temperature. The tested samples coating thickness was of 100 µm, 200 µm and 400 µm. The samples were tested to a sufficient number of cycles so the coating is exfoliated.

The oven is heated at the test cycling temperature. The sample is moved from the environment temperature into the oven. The heating speed of the specimen is variable depending on the specimen size, type of material, single layer or multilayer. The specimen is moved from inside the oven to the cooling area where is cooled till about 40°C.

In Figure 2 are presented images of the samples with the three thicknesses, before and after the thermal shock test.

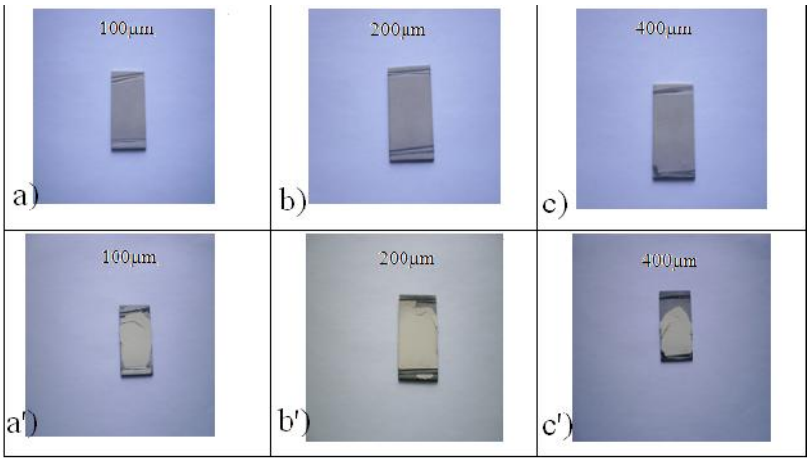

Figure 2: Samples with the coating thickness of 100 µm, 200 µm and 400 µm before and after the thermal shock test at 1200 ºC: a;b;c- before thermal shock test; a';b';c'- after thermal shock test at 1200°C

In Figure 3 is shown the resulting chart after the thermal shock test at a temperature of 1200 °C, and in Figure 4 the images captured during the heat shock test using the Lab View software for the sample with the coating thickness of 100 µm.

The parameters used for the rapid thermal shock test are: the average speed of heating of the sample in the first 10 seconds is which 75,57 °C/s, the average speed of cooling of the sample in the first 10 seconds is which 69,34 °C/s for 60 s of cooling, while maintaining the sample in the oven 5 minutes and the duration of the test in of 6 minutes. The maximum pressure of the compressed air during cooling is of 8,7 bar and the minimum pressure has the value of 7,13 bar.

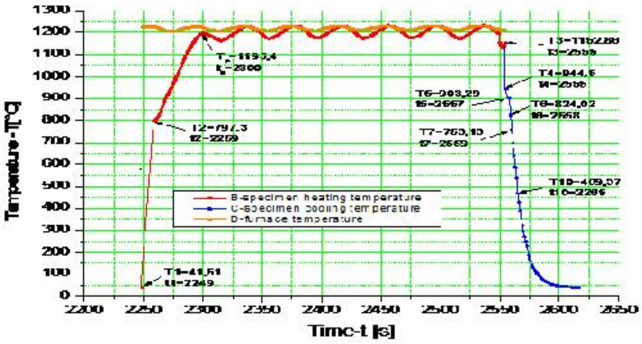

Figure 3: The chart resulted from the thermal shock test at 1200 ºC for the sample with the coating with the thickness of 100 µm

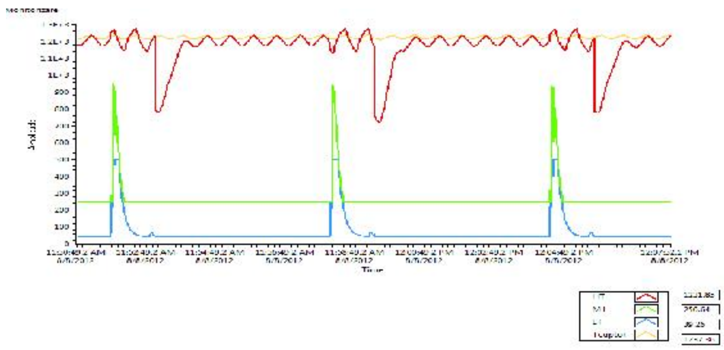

Figure 4: Images captured with the Lab View software during the thermal shock test

Following the thermal shock test electron microscopy investigations were performed and compared to the untested samples in order to observe the structural changes that occurred.

In Figure 5-a is presented a SEM cross-section image of a blank sample where the good adhesion between coating and substrate can be observed. Following the heat shock test at the temperature of 1200 ºC on the sample a crack was identified at the interface between coating and substrate (Figure 5-b) [2].

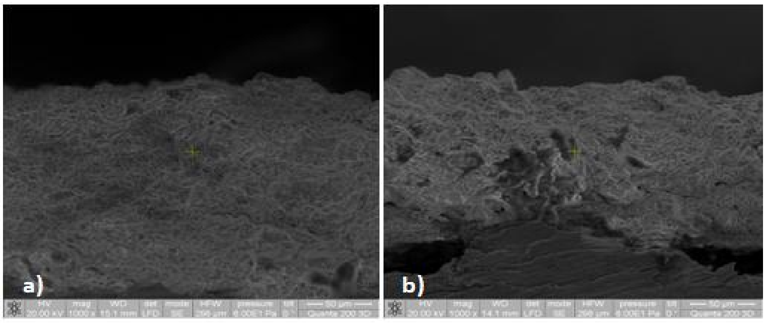

Figure 5: SEM images in cross-section of the YSZ coating:
a) before the thermal shock test; b) after the thermal shock test

Making comparison between the microstructure of the deposited layer in cross-section on the witness sample and the one of the sample subjected to thermal shock at the temperature of 1200 °C, it can be concluded that a sintering of the splats had occurred due to temperature (Figure 6-a/b).

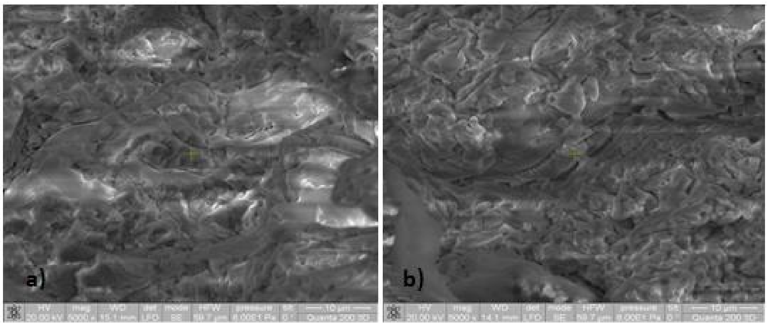

Figure 6: SEM images in cross-section of the YSZ coating:
a) before the thermal shock test; b) after the thermal shock test.

In Figure 7 is shown the chart after the thermal shock test at a temperature of 1200 °C and in Figure 8 the images captured during the heat shock test using the Lab View software on the second sample with the thickness of 200 μm.
The parameters which were used on the installation for the thermal shock test are: the average speed of heating in the first 10 seconds of the sample is which 70,25 °C/s, the average speed of cooling of the sample in the first 10 seconds is which 69,52 °C/s and the time of cooling is which 60 s. The maintaining time in the oven of the sample is 5 minutes. The test duration is 6 minutes, the highest pressure of the compressed air during the cooling process is 8,7 bar and the minimum pressure is 7,13 bar.

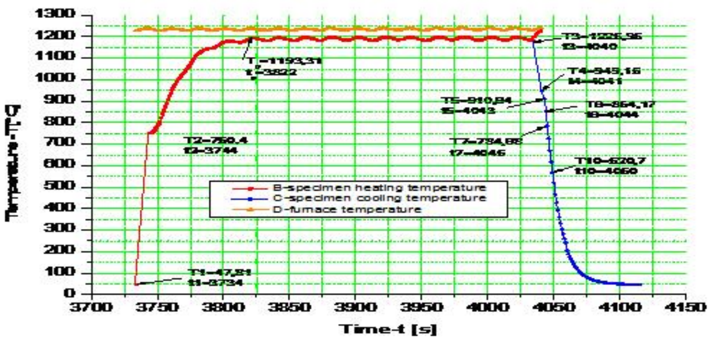

Figure 6: The resulted heat shock graph conducted at a temperature of 1200 ° C for the sample with the coating thickness of 200 μm

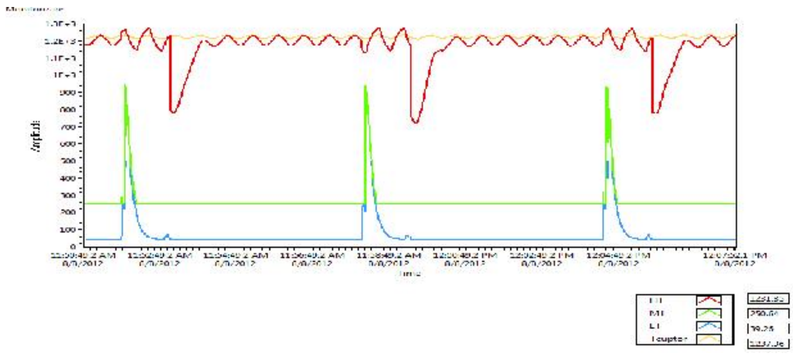

Figure 7: Lab View captured image during the thermal shock test

In Figure 8-a are presented the performed investigations using electron microscopy on the sample with the ceramic layer with a thickness of 200 μm. It can be seen from the SEM cross-section image of a blank sample the good adhesion of the layer with the substrate [3]. Following the thermal shock test a crack appeared at the interface between coating and substrate of about 50 mm, Figure 8-b. The columnar structure observed in Figure 9-b indicates the preferred growth direction of the splats after the thermal gradient direction formed during the thermal shock test cycles.

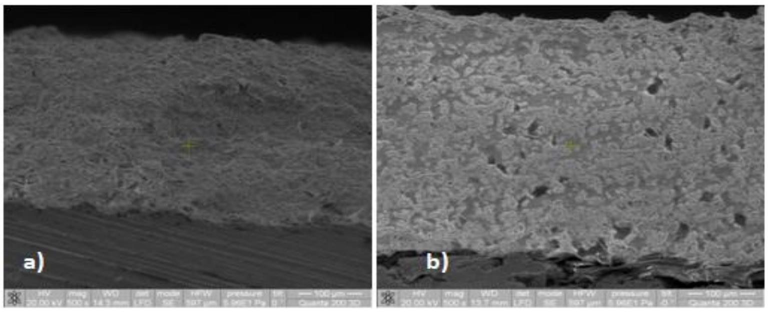

Figure 8: SEM images in cross-section of the YSZ coating:
a) before the thermal shock test; b) after the thermal shock test.

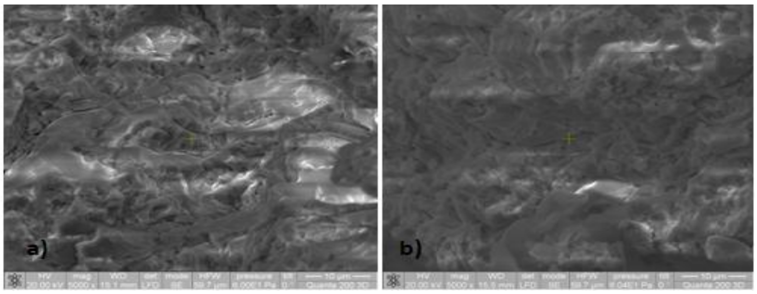

Figure 9: SEM images in cross-section of the YSZ coating:
a) before the thermal shock test; b) after the thermal shock test.

Figure 10 shows the chart after the thermal shock test at a temperature of 1200 ° C and in Figure 11 the image captured during the heat shock test using the Lab View software on the third sample.
The parameters used for the thermal shock test are: the average speed of heating of the sample in the first 10 seconds which is 69,76 °C/s, the average speed of cooling of the sample in the first 10 seconds which is 60,52 °C/s and the time for cooling which is 60 s. The sample was maintained in the oven for 5 minutes and the test lasted 6 minutes. The highest pressure for the compressed air used for cooling is 8,7 bar and the minimum pressure is 7,13 bar.

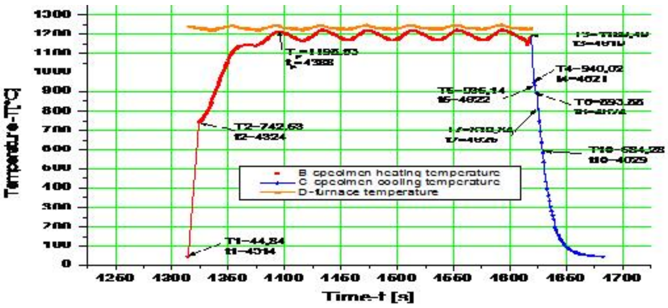

Figure 10: The graph resulted from the heat shock test conducted at a temperature of 1200 ° C for the sample with the coating thickness of 400 µm

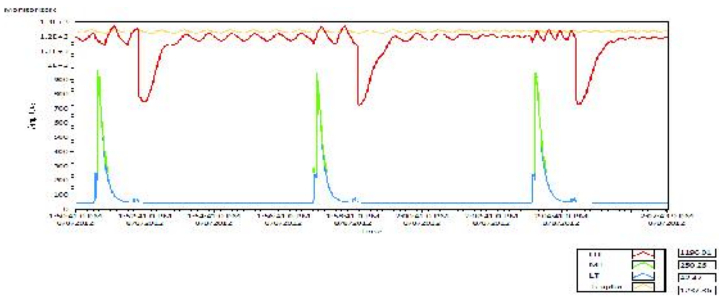

Figure 11: Lab View image captured during the thermal shock test

In Figure 12 are images of electron microscopy investigations of the sample with the coating thickness of 400 µm subjected to the thermal shock test compared with the control sample.

In Figure 12 shows the SEM cross-section image of the blank sample with the coating thickness of 400 µm. The good adhesion of the layer with the substrate can be observed from the picture. Following the heat shock performed at a temperature of 1200 °C, in the sample a crack parallel to the surface of the substrate can be identified which can also be caused by cutting of the sample (Figure 12-b). The crack opening is about 100 µm. Also a diffusion between the ceramic layer and the substrate may also be observed.

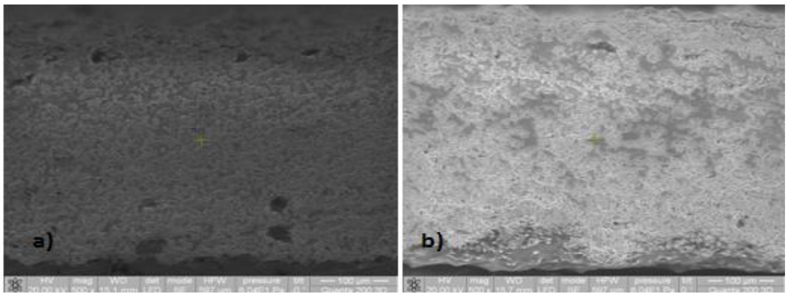

Figure 12: A SEM cross-sectional image which shows the microstructures of the YSZ surface layer: a) before the heat shock test, b) after the heat shock test at a temperature of 1200 °C.

Because of the surface diffusion between the two deposited layers, isolated areas can be observed with splats which are specific to the ZrO_2/20% Y_2O_3 ceramic layer (Figure13).

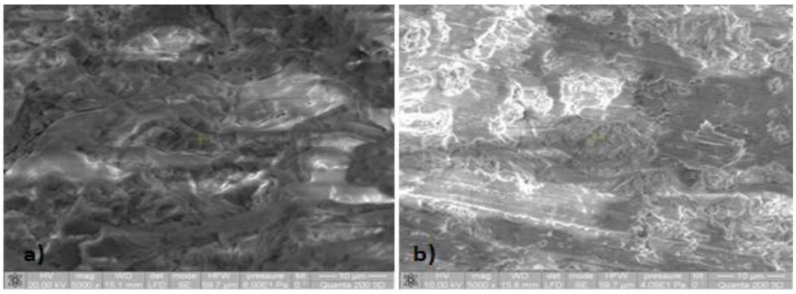

Figure 13: A SEM cross-sectional image which shows the microstructures of the YSZ surface layer: a) before the heat shock test, b) after the heat shock test.

4. CONCLUSIONS

It could be concluded that after the thermal shock test for the three samples the one with the coating thickness of 400 µm behaved better for both heating and cooling than the other two layers with different thickness, due to a better insulation. Also the samples with the coating thickness of 100 µm and 400 µm lasted for 14 test cycle in comparison to the one with the thickness of 200 µm which lasted for only 11 cycles.Taking into account the operating conditions of the gas turbo engines in terms of thermal shock a coating with the thickness of 400 µm is more effective.

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