THE ADHERENCE OF A ZrO₂/20%Y₂O₃ CERAMIC COATING ON A Ni BASE SUPER ALLOY FOR TURBINE BLADES APPLICATION

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Abstract: Atmospheric plasma spraying (APS) is a widely used deposition method for obtaining variable thickness coatings on surfaces with different degrees of complexity. This paper presents a concept of thermal barrier coating which consists of a $ZrO_2/20\%Y_2O_3$ ceramic top layer and a Ni-22wt%Cr-10wt%Al-1wt%Y bond layer, both deposited by atmospheric plasma spraying (APS) on a Ni based super alloy. The analyzed samples were obtained by thermal deposition with the METCO 7 MB plasma jet installations. The paper analyzes the adhesion of the deposited coating on the base material. The adherence test was performed using the "scratch" method with UMTR 2M-CTR type tribometer. To assess the surface roughness measurements were conducted using Form Talysurf Intra system, manufactured by Taylor Hobson LEICESTER, ENGLAND. Highlighting and interpretation of the results were performed using scanning electron microscopy with Quanta 200 3D DUAL BEAM electron microscope.

Keywords: ZrO₂/20%Y₂O₃, scratch, adhesion

1. Introduction

The deposition of a ceramic layer on a Ni base super alloy aims to achieve a thermal barrier for the base material which has a great importance for the manufacturing of turbine blades used in aircraft turbine engine.[1,3]

The obtained quality for thermal spray coatings can be assessed by: adhesion, roughness, thickness, strength, porosity.[2,5] These aspects can be influenced by the technological parameters used for the adopted spraying methods.

Another important aspect which gives different layer properties is the material used to obtain de deposited layer.[4,5] This paper analyses the way how a $ZrO_2/20\% Y_2O_3$ layer, deposited by plasma jet method, behaves in terms of adherence on a Ni based super alloy.

Adhesion is a molecular or atomic interaction phenomenon acting on the contact area between two solid state material surfaces.[7,10]

This process is obvious when one of the two solid layers in contact is thin or has a small size. When the contact area of the surfaces is large, the real contact points will represent only a small part of the total area, therefore the specific adhesion of these points will be weak, related to the entire surface of the bodies. [9]

The materials plasticity has an important influence on adherence for materials in contact. With the increase of plasticity, the adhesion between the layers will be stronger.[11] The thermal state of the contact materials is another crucial factor to obtain a strong adhesion between layers. [12]

2. Materials, methods and instrumentation

The samples used for the scratch test were composed of a Ni super alloy base material on which a successive deposition was made using atmosphere plasma jet with a 7MB METCO type installation. The materials used for the deposition were a NiCrAlY bond material and $ZrO_2/20\% Y_2O_3$ as the top coat.

The coating material is produced by SULTZER METCO in the form of a powder. Because of the small adhesion strength between the deposited ceramic layer and the base material it is recommended to use an intermediate layer. Therefore we used Amdry 962 (NiCrAlY) powder as intermediate layer. The powder has excellent resistance to oxidation and corrosion at temperatures above 1050°C (1323 K).

The commercial denomination of the ceramic powder used for plasma spraying is Metco 202NS ($ZrO_2/20\% Y_2O_3$). This type of powder is widely appreciated because of the good behavior as insulator and very good resistance to thermal wear at temperatures above 980°C (1253 K). In Table 1 are presented the parameters used for the plasma jet deposition.

Table 1:	Parameters	of deposition
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Technological	NiCrAlY	ZrO ₂ /
parameters		20%Y ₂ O ₃
Spray distance, (mm)	120	120
Injector	1,8	1,8
Plasma gas intensity, (A)	600	600
Arc voltage (U)	62	65
Speed of rotation	55	55
(rot/min)		
Argon flow (m^3/h)	50	40

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 (Fig.1).



Figure 1: Quanta 200 3D electron microscope

The scratch test conducted on the UMTR 2M-CTR type tribometer uses three methods to detect the coatings failure. Firstly, a load cell is used to measure the change of the friction force value, secondly the acoustic emission made by the cracks are detected and thirdly, after the test is complete, the scratch channel can be viewed using an optical or an electron microscope, to see how the crack has propagated.

The intensity of the acoustic emissions is dependent on the type of failure suffered by the

coating during the adhesion test: cracking, chipping or delaminating.

For this reason is important to view how the coating had failed after the adhesion test and to confirm the critical load by analyzing the failure with a microscope.



Figure 2: CETR UMT-2 tribometer

The method used to assess the analyzed layer in this paper is the progressive loading method (PLST - Progressive Load Scratch Test) and consists of applying a force gradually (from 0 - 19N) over the indentation for a defined period of one minute. The speed of the indentation is 10 mm/min. The indenter used is a DFH-20 Dual Friction/Load Sensor, which has been mounted with a micro blade with a peak radius of 0.4 mm.

By varying the spraying parameters different thicknesses of the TBC deposition coating are achieved. The thicknesses are 100 μ m, 200 μ m and 400 μ m.

3. The scratch method used to determine the adhesion of the layer

There are three characteristic zones of the scratch indentation: the initiation zone (corresponding to the application of force from 0 N to 7 N), an intermediate zone (7-13 N) and the final (13 to 19 N). These zones were used to observe the behavior of the coating at progressive loading. After the scratch testing on the first samples it can be observed that the destruction of the layer starts from the final zone (corresponding to approximately 13 N of force after approximately 38 sec.). This can be seen in Fig. 3 on the SEM images and especially using the energy - dispersive X-ray spectroscopy performed on the final scratch, as can be observed in Fig. 4.

The third analysis performed is drawing the profilometry of the final segment of the scratch (Fig. 5). It can be seen that the destruction of the layer has a width of 1.2 mm and a depth of 185 μ m.

By using the data from the chart presented in Fig. 6, calculations were carried out from which resulted that the exfoliation begun in the 38 second which corresponds to a force of 13 N.

On the same chart high variation of the friction coefficient are observed. The maximum value of the friction coefficient is 0,179. The variation of the friction coefficient is due to the porosity of the layer and the columnar stratified structure of the material. The friction coefficient drops to the value of 0,04 when the indenter reaches the base material.



Figure 3: SEM imagines of the scratch marks on the sample with the layer thickness of $100\mu m$: a) initial zone (0 - 7 N), b) intermediate zone (7 - 13 N) and c) final zone (13 - 19 N)



Figure 4: Spectral analyses of the final mark of the scratch on the sample with the layer thickness of $100\mu m$



Figure 5: The profile of the scratch for the $100\mu m$ layer sample



Figure 6: The variation of the friction coefficient due to the progressive applied force

For the sample with the layer thickness of 200 μ m in Fig. 7 is observed that the coating was not affected visibly after the scratch test. The present three zones are: the initiation zone (corresponding to the application of force from 0 N to 4,5 N), an intermediate zone (4,5-14 N) and the final zone (14 to 19 N). The fact that no visible damage to the layer is produced is proved also by the spectral analyses from Fig. 8 on which only chemical elements specific to the layer are present: Zr, Y.



Figure 7: SEM imagines of the scratch marks on the sample with the layer thickness of $200\mu m$: a) initial zone (0 - 4,5 N), b) intermediate zone (4,5 - 14 N) and c) final zone (14 - 19 N)



Figure 8: Spectral analyses of the final mark of the scratch on the sample with the layer thickness of 200µm

However, on the scratch profile presented in Fig. 9 a mark can be seen with a width of 0,8 mm and depth of $100 \ \mu m$.



Figure 9: The profile of the scratch for the 200µm layer sample

On the chart resulted from the scratch test (Fig. 10) can be observed that in the first 15 sec the friction coefficient is high due to the pronounced porosity and after that it's decreases. This happens due to the smoothing of the coating. After the smoothing of the layer the friction coefficient value is around 0,03.



Figure 10: The variation of the friction coefficient due to the progressive applied force for the sample with coating thickness of $200 \ \mu m$

For the sample with the coating thickness of 400 μ m the behavior is similar to that of the coating with the coating thickness of 200 μ m. In Fig. 11, which presents the SEM image of the coating, no notable marks can be seen (in all of the three zones of the test).

The integrity of the layer is also highlighted by the elementary chemical analysis which presents only the chemical elements specific to the layer.

On the scratch profile the indenter mark has the following dimensions: a width of 0,8 mm and a depth of 200 μ m (Fig. 13).



Figure 11: SEM imagines of the scratch marks on the sample with the layer thickness of 400μ m: a) initial zone (0 - 5 N), b) intermediate zone (5 - 13 N) and c) final zone (13 - 19 N)



Figure 12: Spectral analyses of the final mark of the scratch on the sample with the layer thickness of $400\mu m$



Figure 13: The profile of the scratch for the $400\mu m$ layer sample

In the first 12 sec of the scratch test, for the third sample, the friction coefficient is also high due to the porosity of the coating (Fig. 14.). With the increase of the applied force a smoothing of the

layer happens so the friction coefficient decreases at a average value of 0,04.



Figure 14: The variation of the friction coefficient due to the progressive applied force for the sample with coating thickness of $400 \ \mu m$

Conclusions

The paper followed the influence of the deposited ceramic layer thickness concerning its properties and characteristics. To obtain different coating thicknesses, the number of passes of the spraying gun on the working surface was varied.

The coating adhesion testing was performed with the scratch method. The obtained results were conclusive regarding the adhesion of the layer and its minimal thickness. The samples with the layer thickness of 200 and 400 μ m had the best adherence to the base material, while the sample with the coating thickness of 100 μ m exfoliated in the final zone of the test. Given the harsh thermal and stress conditions in which turbine blades operate it is very important for the protecting ceramic coating of the layer to have a good adherence to the base material. In this respect the deposited coatings with thicknesses of 200 and 400 μ m are recommended for use on turbine blades.

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