

INNOVATIVE CHARACTER OF PLASMA SPRAY DEPOSITION METHOD, AT THE CERAMICS POWDER Al_2O_3 - TiO_2

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Abstract: *The Atmospheric plasma spraying method (APS) is one of most used methods for deposition on layers with different thickness on surfaces with different degrees of complexity.*

This paper presents a new concept of thermal barrier coating for the prevention of delaminating of the sprayed layer and this consists of an adherent layer of Ni Mo Al (90-5-5) and a top coat of Al_2O_3 - TiO_2 , deposited by atmospheric plasma spraying (APS) on specimens of nodular cast iron of which the camshaft are made of the internal combustion engine

The samples studied in this paper were obtained by thermal spray using an atmospheric plasma spray installation SPRAYWIZARD - 9MCE.

To highlight results, SEM (Scanning Electron Microscopy) analyses were performed using a Quanta 200 3D Microscope Dual Beam. Using x-ray analysis it was possible to detect the existing phases and constituents in the layer deposited with the plasma spray method and comparisons were made about the composition of the used powders. Testing adhesion and cohesion of the coating by the "scratch" was made with micro tribometer UMTR 2M-CTR.

Key words: Al_2O_3 - TiO_2 , SEM, XRD

1. Introduction

Camshafts are trained by the engine crankshaft by means of a needle belt (distribution on the belt) or a metal chain (distribution chain). The shafts are made of lightweight steel alloy or alloyed iron.[1]

The contact between the cam and cam followers is always lubricated with engine oil. Because each valve opens once a complete engine cycle (two revolutions of the crankshaft), camshaft speed is half that

of the crankshaft. The main applications of shaft distribution are given essentially by bending, torsion and the crushing, at the level of cams.

These operational conditions require increased rigidity of the shaft distribution, so there can not appear dangerous deformations.[2] Equally, the cams must have a hard surface and a profile to ensure optimal gas exchange, with adequate reliability.

This paper approaches hardening the

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cams mechanism distribution using deposited by atmospheric plasma spraying , using ceramics powder $Al_2O_3-TiO_2$. [3]

2. Experimental procedure

This paper presents a new concept of thermal barrier coating for the prevention of delaminating, spalling and surface distress of the sprayed layer. This consists in an adherent layer of Ni Mo Al (90-5-5) sprayed with electric arc and a top coat of $Al_2O_3-TiO_2$ deposited by atmospheric plasma spraying (APS) with SPRAYWIZARD-9MCE by Sulzer Metco. The samples before being sprayed were cleaned in an ultrasonic bath with acetone and sand blasted with electro corundum.

To highlight the results analysis were performed using electron microscopy with electron microscope QUANTA 200 3D DUAL BEAM and X-Ray diffraction was performed using an X' Pert PRO MRD equipment. Testing adhesion and cohesion of the coating by the "scratch" method was made with micro tribometer UMTR 2M-CTR.

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	$Al_2O_3-TiO_2$
Cooling water debit	8,7 bar
Velocity of rotation	55 rot/min
Electrode voltage (U)	60 V
The intensity of the gas Plasma (A)	600 A
Composition of plasma	46,1% Ar/13,51% H_2
Spraying distance	120

Technical parameters Table 2

Smart Arc 350	Ni Mo Al
U	31V
I	200A
Air pressure	60 PSI

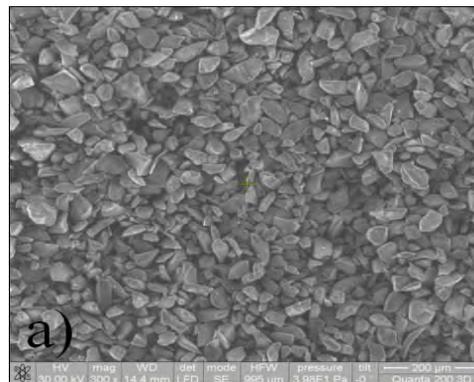
3. Experimental results

3.1. Micro structural characteristics

The analyses presented below were performed using microscopy with electron microscope Quanta 200 3D type. Working in High Vacuum mode (the pressure of the order of 10^{-4} Pa), the sample being attached on the specific support with bands using a special carbon, and the EDS analysis was performed using EDAX – AMETEK, attached on the same scanning electron microscope.

In Figure 1 the morphology of the powder used and its chemical composition is presented. To be certain that the powder is melting in the plasma jet, for a given set of spray parameters, the powder size must be checked, because the quality of coverage depends on it. [3]

If powders are not made at the necessary dimensions for the spraying parameters, the quality of the coating starts to deteriorate.



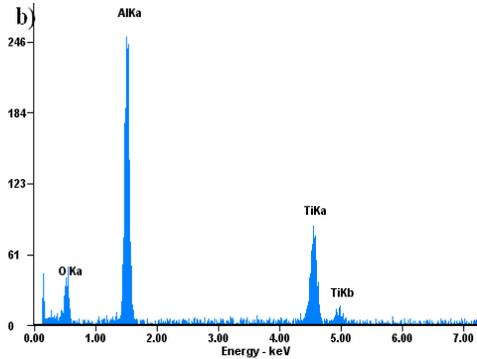


Fig. 1. Powder $Al_2O_3-TiO_3$: a) SEM image of the powder used and b) the EDS chemical analysis.

In order to highlight all the chemical elements on the surface layer, we made the elementary distribution map of chemical elements present in the layer. (Fig.2.a/b).

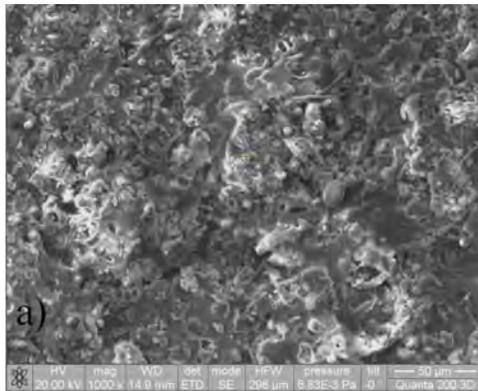


Fig. 2. a) Surface observations for $Al_2O_3-TiO_3$ coatings and b) the distribution map of the chemical elements on ceramic layer $Al_2O_3-TiO_3$

In Fig.3. coating layer is characterized as having a lenticular structure, specific for layers deposited by plasma spraying, highlighting that the splat structure has a smaller structure from a dimensional point of view. In contrast, even if the splat can be seen well defined after fracture, those areas in between that lack adhesion cannot be seen, layers being more compact and homogeneous.

To observe the lamellar nature of the layer a sample was cut off from the part on which the layer was deposited

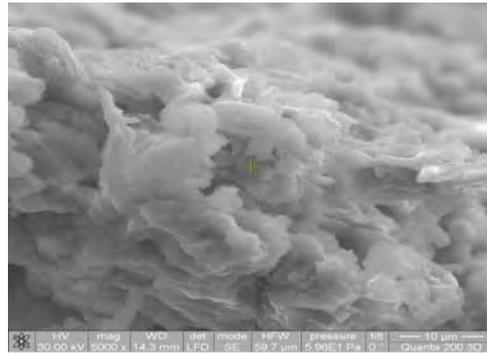
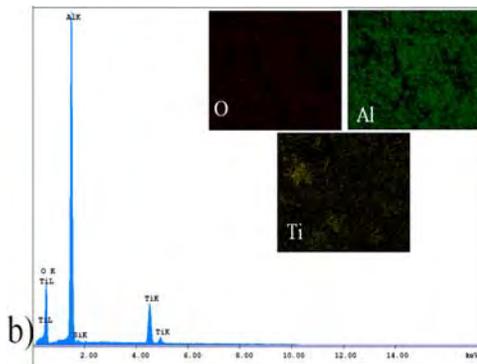


Fig. 3. Micro structural observations along the cross section

Also using the electron microscopy, layer measurements were made, the thickness determined values ranging from 40 to 60 μm, as shown in Figure 4.



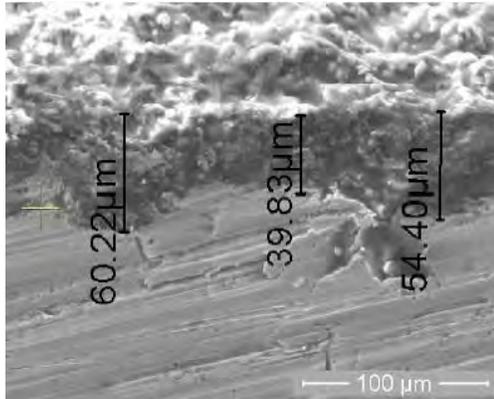


Fig. 4. SEM image for the layer thickness

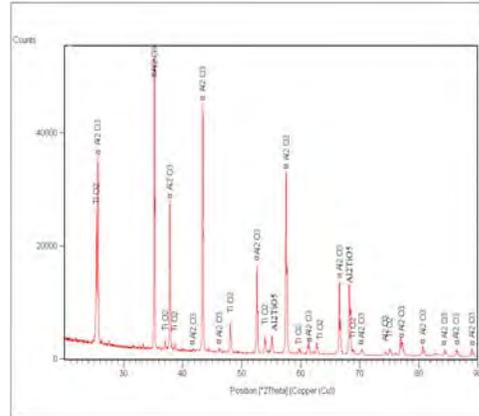


Fig. 5. XRD spectrum of the powder (Al_2O_3 - TiO_3)

3.2. X-ray diffraction

Using XRD analysis, phases and constituents present in the coating obtained by plasma spraying have been observed, compared with the powder used. These phase transformations may help to understanding the behaviour of the layers at different types of testing. The operating configuration for X'PERT PRO MRD diffractometer, with which analysis were performed are: Copper anode with $\lambda = 1.54 \text{ \AA}$, supporting samples open eulerian cradle, the samples were run at 2θ from 20° to 90° .

The XRD analysis (Figure 5) of the powder used revealed its composition: α Al_2O_3 (corundum) and γ Al_2O_3 approximately equal in proportions, TiO_2 (under the form of anatase and the rutile). The general composition of the crystalline ceramic layer obtained, that can be observed from the diffractometry shown in figure 6, shows that most crystalline phases are: α Al_2O_3 (corundum), TiO_2 (such as anatase - majority, as well as rutile), but also a newly formed phase - Al_2TiO_5 - complex molecule.

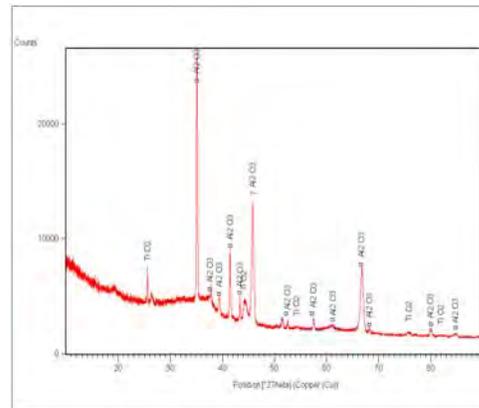


Fig. 6. XRD spectrum of the APS-sprayed coating (Al_2O_3 - TiO_3 layer)

High proportion of α Al_2O_3 can be explained considering that during the application process powder is melted in the plasma spray with formation of γ Al_2O_3 phase, but when the particles reaching the substrate are cooled sufficiently slow (due to substrate heating) the transformation of the whole quantity of α Al_2O_3 in γ Al_2O_3 takes place.

4. Adhesion and cohesion testing for sprayed coating using the "Scratch"

The necessity of evaluating the adhesion

and the cohesion of a coating at the substrate comes from the close connection between them and the wear and abrasion resistance, because the damage caused by them have their origins in the deposited layer.

The method used to assess the layer analyzed in this research is the progressive loading one (PLST - Progressive Load Scratch Test) and is achieved by applying a force gradually (from 0 - 19N) over the indentation for a defined period - 1 min, the forwarding speed of the indentation being 10 mm/min using UMTR micro tribometer 2M-CTR.

The indentation used is a DFH-20 Dual Friction/Load Sensor, which has been mounted with a micro llama with a peak radius of 0.4 mm.

In order to evaluate the results, the sample was analyzed using SEM images, chemical analysis and profilometry of the scratch. In Figures 7 a / b visible damage of the layer cannot be seen but only a trace left by friction between layer and indenter (in both parts).

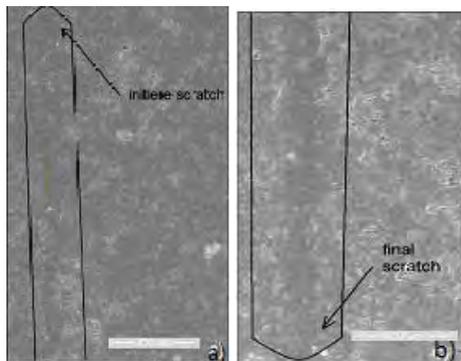


Fig. 7. SEM image of the scratch: a) starting area (0 - 4N), b) final area (15 - 19N)

Layer integrity is highlighted by the chemical elements distribution map in Figure 8 where only sprayed layer specific elements are present and any other

chemical elements cannot be observed (which could come from the substrate).

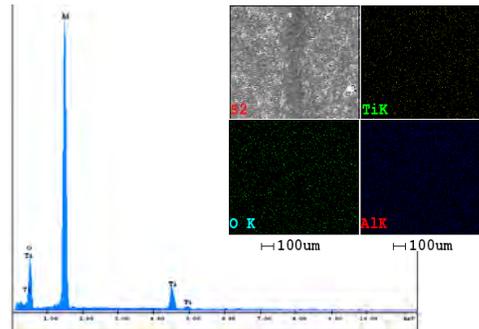


Fig. 8. Map distribution and spectral analysis of elements on area final scratch (15-19N)

However, in Figure 9 is presented the profilometry of the scratch and a deterioration by a slight impression of the sprayed layer is observed (a width of approximately 243 µm, and a depth of approximately 5 µm).

On the resulting graph after the scratch test (Figure 10) is observed the increase of normal force with load and large variations of friction and friction coefficient (but is not greater than 0.1), most likely caused by the pronounced porosity of the deposited layer.

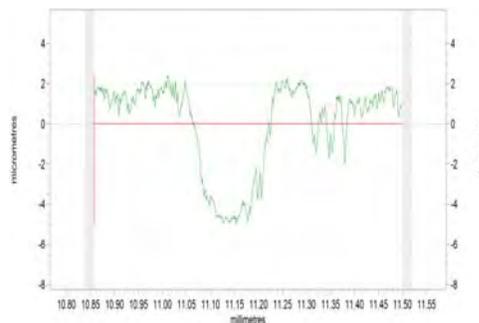


Fig. 9. Profile of the final area scratch on the sample

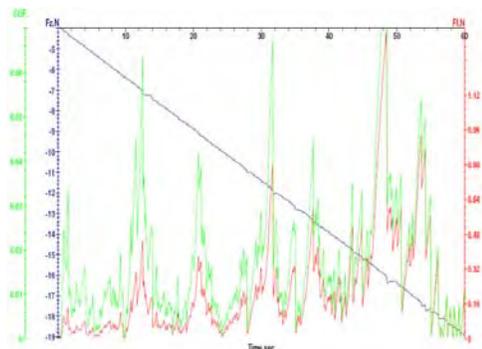


Fig. 10. Graph generated during the scratch test on the sample

Summary

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 of the Al_2O_3 - TiO_2 layer with a maximum 60 μm thickness.

Characterization of the layer obtained was performed by several methods: with electron microscopy were made remarks on the layer thickness and its microstructure, using X-ray diffraction have been analyzed phases and constituents, and using UMTR 2M-CTR micro tribometer, adhesion and cohesion test was performed.

Using electron microscopy a good stiffness and also a good substrate adhesion to support the filler material layer was observed.

By X-ray diffraction analysis, comparison are performed on the powders used and the layers obtained from them and different peaks were revealed featuring the following phases: a) α Al_2O_3 (corundum) and γ Al_2O_3 approximately equal in proportions and b) shows that most crystalline phases are: α Al_2O_3

(corundum), TiO_2 (such as anatase - majority, as well as rutile), but also a newly formed phase - Al_2TiO_5 - complex molecule.

Testing the cohesion and the adhesion of the coating using the scratch method yielded satisfactory compartment results, with the appearance of small cracks in the final segment of all the scratch (in the area where maximum load was applied).

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