INFLUENCE OF HEAT TREATMENT ON THE MICROSTRUCTURE AND PHASE COMPOSITIONS OF DETONATION NANOSTRUCTURED ALUMINA COATINGS

Maria ARSEENKO¹, Marina KOVALEVA¹, Yurii TURIN², Ivan PAVLENKO¹

¹ Belgorod State National Research University, kovaleva@bsu.edu.ru
² E.O. Paton Electric Welding Institute NASU, y.n.tyurin@rambler.ru

Abstract

In this study the effect of heat treatment on air on the microstructure and phase compositions of nanostructured Al₂O₃ coatings, obtained by multi-chamber gas-dynamic accelerator, was investigated using OM, SEM, TEM with diffraction and XRD techniques. Powder AMPERIT® 740.0 Al₂O₃ was used to form a dense ceramic layer on flat specimens of hot-rolled carbon steel (ASTM A570-36). MCDS has provided the conditions for formation of a dense ceramic layer with porosity below of 2 %. The coatings are consisted of discrete deformed particles (50 - 3000 nm) of oxide and had the lamellar structure. The area of the coating that adjoins to the substrate contains a transition layer of intermetallic compounds up to 15 μm thick. Post-deposition heat treatment of the samples at a temperature of 850°C for 3 h was carried out in an electrical muffle furnace on air. It was established that the heat treatment led to more significant increase in the fraction of α-Al₂O₃ phase component as well as remarkable reduction of porosity.

Keywords: gas-dynamic accelerator, Al₂O₃ coatings, heat treatment

1. INTRODUCTION

Coatings made of the Al₂O₃ powder are widely applied to protect surfaces of parts operating in aggressive environments and at high temperatures. Such wide use is accounted for the high service properties of the material and its low cost [1]. Ceramic Al₂O₃ deposits are characterized by lamellar structure [2]. A fraction of porosity from several percent up to 20% can be formed in coatings [3], which results from the insufficient filling and incomplete wetting of molten liquid on previously formed rough coating surface. The voids in the deposit will influence many deposit properties such as mechanical and physical properties [4]. The pores are constitutive of large voids of a size from several micrometers up to over 10 μm and two-dimensional small voids including the nonbonded interface between lamellae and vertical cracks in individual ceramic lamellae [5]. It is well known that the structure and properties of thermally sprayed coatings usually differ from those of corresponding wrought or cast materials. This is caused by the specific coating formation mechanism, which causes the presence of various levels of residual porosity, cracks, interlamellar voids, oriented microstructure and chemical inhomogeneity in the sprayed coatings. In most cases, these voids are undesirable and are tried to be avoided by means of selection of appropriate spray technique, optimisation of the deposition process and the properties of the material to be sprayed. Although thermally sprayed coatings are frequently used in their as-sprayed condition, or after finishing by machining or grinding, there remains often needs to carry our various post-treatment procedures before the use of the coatings. These post-treatments are typically different heat-treatments, mechanical treatments or filling the residual porosity with sealants. Main objects are to improve mechanical properties or to provide corrosion protection of the substrate and the coating material [6]. Heat treatment is the usually applied process in post-spraying treatment of thermally sprayed coatings. Post-spray treatment has been widely recognized in the last decade as the key to the quality of coatings [7]. Post-treatment of thermal spray coatings has been shown to refine the microstructure e.g. by eliminating porosity and microcracks, and to promote beneficial phase transformations and metallurgical bonding within the coating microstructure [8]. In this study the effect of heat treatment on air on the microstructure and phase compositions of nanostructured Al₂O₃ coatings, obtained by multi-chamber gas-dynamic accelerator, was investigated using OM, SEM, TEM with diffraction, SPM and XRD techniques.
2. EXPERIMENTAL

2.1 Coating preparation

Powder AMPERIT® 740.0 Al₂O₃ was used to form a dense ceramic layer on the plate of steel. The powder consisted of crushed particles with a maximal size of up to 50 μm (5-10%), the main fraction being 5.6-22.5 μm, the phase composition is mainly represented by γ-Al₂O₃ (92.34%), α - Al₂O₃ (3.83%), SiO₂ (3.83%) (Fig. 1). Flat specimens of hot-rolled carbon steel (Fe-0.25C-0.90Mn-0.04P-0.05S-0.20Cu, all in wt pct) were used as substrates and they were sandblasted using alumina grits 25A F360 prior to spraying. The dimensions of the samples were of 30 x 30 x 5 mm. The sample was prepared by grinding with abrasive SiC paper (gradation 200, 500, 800, 1000), followed by polishing with 1 μm diamond slurry according to the procedure recommended by Struers company for ceramic coatings.

![Fig. 1 Morphology (SEM) (a) and the XRD pattern of the Al₂O₃ powder (b)](image)

A multi-chamber gas-dynamic accelerator (Fig. 2) [9] with a nozzle length of 500 mm was employed to deposit the powder coating of aluminum oxide on the steel substrate in this study. The automated equipment (Fig. 2) consisting of 1 - a multi-chamber detonation sprayer (MCDS), 2 - standard powder feeder with a feed rate of up to 3 kg/h, 3 – a standard low-pressure (max. 0.3 MPa) gas panel for feeding oxygen, propane-butane and air, 4 – an automated control system for the technological process, 5 – an automated manipulators for moving MCDS and 6 – a specimen holder.

![Fig. 2 Equipment for deposition of coatings using MCDS](image)

2.2 Coating Post-treatment

The heat-treatment of coatings to examine the effect of heat treatment temperature on coating phase structure, microstructure and microhardness was carried out on air using furnace LT 5/12/B180 Nabherthm GmbH. The samples were placed in the furnace and heated at 5°C/min from a temperature of 23°C to 850°C. The coatings were held for three hours at 850°C, before being cooled at a rate of 1.5°C/min until 36°C during the eight hours, and then allowed to cool to room temperature outside the furnace.
2.3 Powder and Coating Characterisation

Examinations of microstructure and elemental composition were carried out by using electron ion microscope Quanta 200 3D, 600 FEG (SEM) and optical microscope Olympus GX 51 (OM). Porosity was determined by the metallographic method with elements of the qualitative and quantitative analysis of the geometry of the pores by using optical inverted microscope Olympus GX51. Local phase and diffraction analysis was conducted by using transmission electron microscope JEOL JEM 2100 (TEM) and X-ray powder diffractometer Rigaku Ultima.

3. RESULTS AND DISCUSSION

3.1 Effect of heat treatment on the microstructure of Al₂O₃ coating

Electron microscopy studies of transverse sections of the «coating - substrate» (Fig. 3a) showed that the coating of Al₂O₃ powder (thickness ~ 200 μm) is characterized by the regular change of packaged "flakes" - deformed discrete particles of oxide. The ceramic layer are observed undeformed fractured powder particles and flaking of large particles during grinding of the samples due to the presence of the starting powder particles sized up to 30 microns (Fig. 3a). The porosity of the Al₂O₃ coating was less than 2%. Coating is formed through successive laying a set of deformed particles that have different temperature, speed and mass (Fig. 3a). The size of the deformed particles was ~ 0.05-3 μm (Fig. 3). The tests of thin foils in the electron transmission microscope have confirmed that the area of the coating that adjoins to the substrate contains a transition layer of intermetallic compounds type of FeAl (FeAl₃, Fe₂Al₅).

![Fig. 3 SEM micrograph of the Al₂O₃ coating (a) as sprayed coating, (b) coating heat treated on air](image)

The porosity of the Al₂O₃ coating after heat treatment on air was less than 0.5% (Fig. 3b). The heat treatment does not lead to changes in transition layer (Fig. 3b).
3.2 Effect of heat treatment on the phase evaluation of Al₂O₃ coating

Figure 5 shows the X-ray diffraction patterns of the alumina coatings. It is obvious that θ-Al₂O₃ is predominant phase in an as-sprayed coating and α-Al₂O₃ is predominant phase in coating after heat treatment. This result may be of considerable interest, especially for dielectric applications.

Fig. 5 XRD patterns of the as sprayed Al₂O₃ coating and heat treated coatings

CONCLUSION

Positive impact of post-coating heat treatment was observed in the microstructure of coatings. It was established that the heat treatment led to more significant increase in the fraction of α-Al₂O₃ phase component as well as remarkable reduction of porosity. The heat treatment does not lead to changes in transition layer.

ACKNOWLEDGEMENTS

This study was supported by Grant of the President of the Russian Federation No. MC-215.2013.8 by using equipment of the Joint Research Center of Belgorod State National Research University.

LITERATURE