ALUMINA NANOSTRUCTURED COATINGS FORMED BY MULTI-CHAMBER GAS-DYNAMIC ACCELERATOR

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Abstract

In this study, multi-chamber gas-dynamic accelerator (MCDS) was applied for deposition of AMPERIT® 740.0 Al₂O₃ powder on flat specimens of hot-rolled carbon steel (ASTM A570-36). The powder consisted of crushed particles with the main fraction being 5.6-22.5 μm. The Al₂O₃ coatings microstructures and phase compositions were characterized using SEM, TEM, OM, SPM and XRD techniques. Measurement of the microhardness of samples was done using a Vickers’s indenter with load on of 0.30 N. It was established that MCDS has provided the conditions for formation of a dense ceramic layer with hardness of 1320 ± 25 HV₀.₃ and porosity below of 1 %. α-Al₂O₃ is predominant phase in coatings. The coatings is characterized by the regular change of packaged "flakes" - deformed discrete particles of oxide and had the classical lamellar structure of thermally sprayed coatings with good lamellar bonding. The size of the deformed particles was ~ 50 - 3000 nm. The area of the coating that adjoins to the substrate contains a transition layer of intermetallic compounds such as FeAl₃, Fe₂Al₅.

Keywords: gas-dynamic accelerator, Al₂O₃ coatings, microstructure, hardness, porosity

1. INTRODUCTION

The coatings of Al₂O₃ powder have drawn great attention for their high strength, hardness, the corrosion resistance and high wear-resistant and have been regarded as potential materials to increase reliability and component products operating at high temperatures, speeds and in harsh environments [1]. Because of excellent properties of alumina ceramics, they are widely used in many refractory materials, grinding media, cutting tools, high temperature bearings, a wide variety of mechanical parts, and critical components in chemical process environments, where materials are subject to aggressive chemical attack, increasingly higher temperature and pressures [2]. Ceramic coatings usually are characterized by a relatively high open porosity which is deleterious when the coatings have to perform in an aggressive environment [3]. There are also adhesion problems between the oxide coating and metallic substrate. A viable solution is to insert a metallic "bond coat" between the substrate and the coating [2]. In this study, the coatings of Al₂O₃ were deposited on STE255 steel substrate by using cumulative-detonation equipment. The microstructure, composition, porosity, hardness and wear resistance of Al₂O₃ coatings were investigated.

2. EXPERIMENTAL

2.1 Materials

Powder AMPERIT® 740.0 Al₂O₃ was used to form a dense ceramic layer on the steel STE255. 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%) (Table 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.

2.2 Apparatus and Procedures

In this work for spraying of powder coating of aluminum oxide a multi-chamber gas-dynamic accelerator was used (Fig. 1) [4]. The Al₂O₃ coatings were carried out using the parameters summarized in Table 1. Nozzle of a throat diameter of 16 mm was adopted.

![Equipment for deposition of coatings using MCDS](Fig. 1)

Table 1 Spraying parameters

<table>
<thead>
<tr>
<th>Flow rate of fuel mixture components, m³/h</th>
<th>Nozzle length, mm</th>
<th>Frequency, Hz</th>
<th>Distance from the sample, mm</th>
<th>Powder feeding, g/h</th>
<th>Speed of powder, m/s, ± 200</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen</td>
<td>500</td>
<td>20</td>
<td>60</td>
<td>720</td>
<td>1300</td>
</tr>
<tr>
<td>Propane + butane</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Air</td>
<td></td>
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</tr>
</tbody>
</table>

*4.16 / **3.55 | *0.8 / **0.54 | *0.25 / **0

*Cylindrical combustion chamber. **Disk combustion chamber

The microstructure and elemental composition of Al₂O₃ coatings are characterized by scanning electron microscopy (SEM) equipped with an X-ray detector of the PEGASUS 2000 system, scanning probe microscope Integra Aura (SPM) and optical microscopy. 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. The hardness tests were carried out with automatic micro-hardness analysis system DM-8 by Vickers’s test applying load 300 g. X-ray diffraction studies of coatings were performed on a diffractometer ARL XTRA. Local structural and phase studies were conducted by using a transmission electron microscope JEOL JEM 2100.

3. RESULTS AND DISCUSSION

Electron microscopy studies of transverse sections of the «coating - substrate» (Fig. 2) 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. 2). The porosity of the Al₂O₃ coating was less than 1-2%. Coating is formed through successive laying a set of deformed particles that have different temperature, speed and mass (Fig. 3). The size of the deformed particles was ~ 50-3000 nm (Fig. 3, 4).
Fig. 2 SEM images of cross section of the Al₂O₃ powder coating: x 500 (a), 5000 (b)

Fig. 3 SEM images of cross section of the Al₂O₃ powder coating: x 10000 (a), 20000 (b), 40000 (c)

Fig. 4 SPM micrograph of the Al₂O₃ coating
High speed of dispersed materials provides their deformation, mechanochemical reaction and the formation of a sufficiently thick (5 μm) of the transition layer. Area of the coating that adjoins to the substrate contains a transition layer of intermetallic type of intermetallic compounds such as FeAl₃, Fe₂Al₅ (Fig. 5).

**Fig. 5** Microstructure and elemental composition of transverse sections of the sample (SEM) (a) and TEM image and diffraction TEM photographs of a transition layer “coating/substrate”

It was established that the coating consisted mainly of θ and α Al₂O₃ an insignificant amount γ-Al₂O₃ and elements of the amorphous phase (Table 1 and Fig. 6).

**Table 2** The phase composition (%) of the Al₂O₃ powder and coating

<table>
<thead>
<tr>
<th>Material</th>
<th>Phase composition, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>α-Al₂O₃</td>
</tr>
<tr>
<td>Powder Al₂O₃</td>
<td>3.83</td>
</tr>
<tr>
<td>Coating Al₂O₃</td>
<td>13.35</td>
</tr>
</tbody>
</table>

**Fig. 6** Results of X-ray analysis of Al₂O₃ coating

**Conclusion**

Examinations of the coating structure showed that was formed of a dense ceramic layer with hardness of 1460±25 HV₀.3 and porosity below 1% These characteristics are comparable with properties of the coatings produced with the agglomerated nano-sized powders by using the HVOF units operating with hydrogen [5]. The absence of any substantial scatter in micro-hardness values from point to point is not worthy. The measured values of micro-hardness are stable throughout the layer (variations are no more than 5 %), which is indicative of the uniformity of the layer of the adhering deformed particles, as well as of the phase and structural uniformity of the coating material. In the transition layer “coating/substrate” were formed the
intermetallics of systems type FeAl, which indicates the high as the compound the substrate with coating. We consider that the coatings of Al₂O₃ powder can be used as material with excellent durability and operating characteristics.

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REFERENCES


