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Deposition and characterization of Al_2O_3 coatings by multi-chamber gas-dynamic accelerator

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ABSTRACT

A series of Al_2O_3 coatings have been prepared on steel substrate by multi-chamber gas-dynamic accelerator. The coatings (200 μm thick) were deposited in 6 passes in the three modes at different nozzle lengths (300, 400, 500 mm) of multi-chamber gas-dynamic accelerator. The Al_2O_3 coatings microstructures and phase compositions were characterised using SEM, TEM, OM and XRD techniques. The hardness tests were carried out by the Vickers method under loads on the indenter equal to 25 and 300 g. The dense ceramic layers with hardness of 1100 ± 100 $HV_{0.3}$ and porosity of less than 1% have been prepared by multi-chamber gas-dynamic accelerator with a nozzle length of 500 mm. The micro-hardness of substrate under the coating to a depth of 200 μm changes from 400 $HV_{0.3}$ to an average hardness of the specimen material equal to 346 $HV_{0.3}$. The intermetallic compound type of FeAl was revealed in the area of the coating that adjoins to the substrate (TEM/electron diffraction).

1. Introduction

The paper presents the results of investigating the properties of ceramic coatings deposited on a steel substrate by using a multi-chamber gas-dynamic accelerator. Coatings made of the Al_2O_3 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 [1,2]. As scientific literature proves, besides the service properties of the coatings there are important parameters characterising the technology such as deposition efficiency, consumption of energy and gases for deposition of a unit mass of a coating, and productivity [3–5].

Analysis of the published information shows that hardness and porosity are most important characteristics of the ceramic coatings determining their performance. Let us consider the main results obtained in previous treatments. The coatings are deposited by using plasma (APS) [6–8], flame (HVOF) [9–13] and detonation (HFPD) [14,15] devices. As a rule, coatings produced by plasma spraying have micro-hardness of up to 950 $HV_{0.3}$ [13]. Ceramic coatings with high values of hardness (1212 ± 72 $HV_{0.3}$, content of α-Al_2O_3 in a coating — 22% [10]) were produced by using the Top Gun HVOF system, under the following conditions: spraying distance — 152 mm, total gas (oxygen + hydrogen) flow rate — 17.6 and 65.6 m3/h accordingly, powder consumption — 0.576 kg/h, and deposition efficiency — 51%. Coatings with hardness of 1350 $HV_{0.3}$ were produced from the agglomerated powders consisting of nano-sized particles by using standard HVOF units operating on hydrogen [12,13]. It is interesting to note that the use of propane in HVOF units led to a dramatic deterioration of quality characteristics of the coatings. Hardness of the resulting coatings went down to 1000 $HV_{0.3}$ [13]. The coatings produced by using the detonation units (HFPD) had micro-hardness of 1073 ± 124 $HV_{0.3}$, which was the best result obtained for this type of equipment with the fuel mixture consisted of propylene + oxygen [14]. It was found that the maximal deposition efficiency is achieved, as a rule, at productivity as low as 0.3–0.4 kg/h. In this case, deposition efficiency for HVOF devices was 68% [10], and for HFPD—61% [15]. As was shown by the analysis, in the HVOF units with hydrogen used as a fuel gas the maximal velocity of the Al_2O_3 particles with a size of −22 + 5 μm was 870 m/s [9], while in the HFPD units the average values of the powder particle velocity was 820 m/s [15].

The technology based on the use of HVOF torches is characterised by the consumption of large amounts of component gas mixture (20–60 m^3/h). To provide efficient cooling, the HVOF torch nozzles and combustion chambers are made from pure copper, the cooling water has a low temperature, and power-intensive (30–50 kW) coolers have to be applied. Because of a high thermal power of the HVOF combustion products it is necessary to set a big spraying distance (250–400 mm), which may lead to some non-uniformity in the coating layer and reduce the deposition efficiency.

For the spraying of coatings we developed a multi-chamber gas-dynamic accelerator [16], where a high flow rate of working gases in the nozzle and, as a consequence, a high velocity of a gas–powder

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flow are provided by the detonation mode of combustion of the fuel mixture consisting of propane + butane, oxygen and air. Thus, the pulsed (frequency 20 Hz) flow of the combustion products acquires a high velocity (up to 2100 m/s) and pressure (up to 3 MPa), which shortens the time of the effect on the nozzle walls and surface treated (1×10^{-3} s). The accelerator has replaceable nozzles (tubes) with a diameter of 16–18 mm and length of 300 to 500 mm, the spraying distance being 40–60 mm.

2. Procedure for the deposition of coatings and materials

Investigations for the optimisation of the ceramic coating deposition parameters were carried out by using the automated equipment (Fig. 1) consisting of a multi-chamber detonation sprayer (MCDS) — 1; standard powder feeder with a productivity of up to 3 kg/h — 2; standard low-pressure (max. 0.3 MPa) gas panel for feeding oxygen, propane–butane and air — 3; automated control system for the technological process — 4; automated manipulators for moving MCDS — 5 and specimen holder — 6.

Characteristic feature of MCDS is that the powder is accelerated by using the combustion products, which are formed in the MCDS chambers and are converged before entering the nozzle, where they interact with the two-phase gas–powder cloud. A standard powder feeder of the Metco Company is used to feed powder to the nozzle. A continuous gas–powder jet is separated into portions and fed to the nozzle by using a special device — gas-dynamic synchroniser. The process of detonation mode of combustion of the fuel mixture (propane–butane, oxygen and air) is initiated in a special chamber by using a spark plug at a frequency of 20–50 Hz. Then the detonation mode of combustion spreads to the other chambers. Such initiation system and powder feeding scheme synchronise both the processes of combustion and the injection of the powder into the nozzle [17].

The temperature and velocity of the powder material are of a decisive importance for the process of formation of a ceramic layer, and consequently the technology developed for deposition of the coatings was implemented in three characteristic modes (Table 1) reflecting the above mentioned parameters.

In mode 1 a fuel mixture diluted with air was used, and the nozzle was 300 mm long. In modes 2 and 3 the fuel mixture contained no diluter, and the nozzle had a diameter of 16 mm and length of 400 and 500 mm, respectively. The detonation initiation frequency was 20 Hz in all cases. Air was used as a powder carrying gas (flow rate — 0.96 m^3/h). The distance from the exit section of the nozzle to the plate treated was 60 mm. The ceramic coatings (200 μm) were deposited in 6 passes in the three modes differing in the nozzle length (Table 1).

The Al_2O_3 ceramic layer is characterised by using scanning electron-ion microscopes Quanta 200 3D and Quanta 600 FEG (SEM) equipped

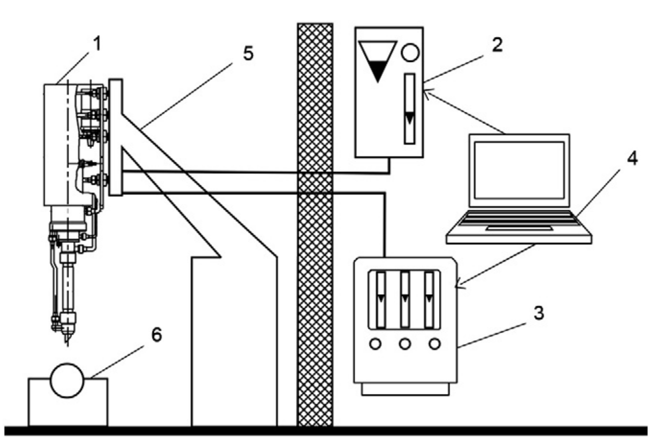

Fig. 1. Equipment for deposition of coatings using MCDS.

Table 1
Modes of formation of ceramic layer from Al_2O_3 powder.

Mode no.	Flow rate of fuel mixture components, m^3/h			Nozzle length, mm	Powder feeding, g/h	Speed of powder, m/s, ±200
	Oxygen	Propane (30%) + butane (70%)	Air			
1	2.27[a]/3.55[b]	0.4[a]/0.77[b]	1.25[a]/1.1[b]	300	550	1000
2	4.16[a]/3.55[b]	0.8[a]/0.54[b]	0.25[a]/0[b]	400	720	1200
3	4.16[a]/3.55[b]	0.8[a]/0.54[b]	0.25[a]/0[b]	500	720	1300

[a] Cylindrical combustion chamber.
[b] Disc combustion chamber.

with an X-ray detector of the PEGASUS 2000 system. Porosity was determined by the metallographic method with elements of the qualitative and quantitative analyses of the geometry of the pores by using optical inverted microscope Olympus GX51 [18]. The images were registered in optic microscope, in the bright field, magnified 500×. The image acquisition of the structure of the studied layer was done with "SIAMS Photolab" programme. At least ten arbitrarily selected typical micrographs were analysed for each experimental point. The hardness tests were carried out with automatic micro-hardness analysis system DM-8 by the Vickers method under loads on the indenter equal to 25 g and 300 g. Indentation was carried out on polished cross-sections of samples in the middle of the coatings with a 20 μm space between the indents. An average of 10 tests was used as an indicator of the coating hardness.

The maximum micro-hardness of layer deposited at Mode 2, was 1500 ± 25 $HV_{0.025}$. In a multilayer coating, obtained in 6 passes, microhardness is the highest on the surface of the coating and gradually decreases the close to the substrate interface, which correlates with a decrease the hardness of the ceramic coating with increasing the number of passes from 1 to 4, noted in [14]. Microhardness measurements at load 300 g have shown a value of 1200 ± 25 $HV_{0.3}$. The difference of values of microhardness readings by almost 300 units is due to the increasing incidence of pores and interlamellar defects on mechanical properties measured over larger length scales. Under higher indentation loads, lamellae slide along their boundaries and microcracks increasingly developed [19]. In the future, the results of the study are only micro-hardness at a load of 300 g.

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 × 30 × 5 mm. The specimens were transversally cut by spark erosion, mechanically polished and prepared by standard metallographic methods — sectioning, mounting and polishing for sample preparation. The sample was prepared by grinding with abrasive SiC paper (gradations 200, 500, 800, 1000), followed by polishing with 1 μm diamond slurry according to the procedure recommended by Struers company for ceramic coatings.

Powder AMPERIT® 740.0 Al_2O_3 (Fig. 2) was used to form a dense ceramic layer on the steel plate (ASTM A570-36) of 5 mm thickness. 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.

Velocity parameters of the gas–powder flow were estimated over a length of 20–80 mm from the exit section of the nozzle by using a two-channel optical measurement system, which comprised two photodiodes FD287 (spectral sensitivity range: 0.5–1.7 μm), plastic optical fibres (POF), amplifiers and a device for real-time recording of signal waveforms. The photodiode spectral sensitivity range and POF spectral window provided filtration of the gas–powder stream radiation by cutting off radiation of combustion products. The boundaries of the heated powder cloud were registered by two measurement systems installed at a distance of 20 mm from each other. The precise time of visualisation in two planes makes it possible to calculate the velocity of the frontal area of the heated powder cloud and its