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The effect of detonation spraying on the phase composition and hardness of Al_2O_3 coatings

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The article were studied the effect of detonation spraying on the structure and properties of Al_2O_3 coatings. It was determined that reducing the delay time between shots is leading to increase the hardness and elastic module of Al_2O_3 coatings. It was found on the basis of X-ray diffraction analysis that the main reason for the increasing in hardness with a decreasing in the delay time between shots is associated with increasing in the volume fraction of α-Al_2O_3 phase. The studies of X-ray diffraction presented that the highest content of the phase is achieved when the coatings are formed with a delay time between shots of 0.25 s. It was found that increasing in the volume fraction of the α-Al_2O_3 phase is caused by the secondary recrystallization $\gamma \rightarrow \alpha$, which occurs due to the heating of particles during coating formation, i.e. due to increasing in temperature above 1100 °C in single spots of the coating when they are put each other.

Keywords: detonation deposition, aluminum oxide, phase, hardness, coating.

Introduction

Exaggeration of operation conditions, complicating the constructions of working elements and components of engineering equipment require the creation of effective means of protection. Most mechanical engineering products are made from steel of various classes, wherein the protection from aggressive external factors is provided by the functional coatings and surface modifications, which is economically feasible [1-3]. In these latter days, there has shown a great interest in high-speed coating spraying technologies, which are characterized by high productivity, versatility, ease of automation, and practically unlimited sizes of coated surfaces. High-speed spraying methods can significantly expand the capabilities of traditional thermal spraying coatings which used to protect parts from deterioration and corrosion [4-7]. The detonation [8], high velocity air-gas plasma (HVAGPS) [9] and high velocity oxygen fuel (HVOF) spraying [10] related to gas-thermal high-speed methods for producing coatings. Among them, the most promising is detonation spraying. Detonation spraying is one of the methods of thermal spraying of coatings, which is carried out by using a special detonation gun filled with explosive gas mixture. A powdery spray material is used to form a coating. The particles of the powder in the process of detonation are accelerated to high speeds (up to 1000 m/s), their melting and deposition on the spraying surface [11, 12]. Special attention is paid to the spraying of composite ceramic, cermet, bioceramic coatings. The detonation method is promising for producing coatings based on Al_2O_3, as well as the high adhesion strength of the coatings due to the low coatings porosity and the chemical composition preservation of the initial powder in the coatings. Al_2O_3 aluminium oxide has a number of positive properties, such as hardness, wear resistance, corrosion resistance, low friction coefficient, and is also an inhibitor of grain growth in metals [13]. The phase composition of the coating from aluminium oxide depends on the application method, process parameters, substrate temperature, size of the sprayed particles and a number of other factors. Traditionally, these coatings are obtained by a flame, plasma or detonation method. It is known [14, 15] that the obtained coating consists practically of γ-Al_2O_3 in case of gas-flame spraying of corundum (α-Al_2O_3), while these are two-phase coatings consisting of α-Al_2O_3 (5-10%) and γ-Al_2O_3 (90-95%) in plasma and detonation spraying. However, available in the literature data on the sequence of phase transformations of γ-$Al_2O_3 \rightarrow \alpha$-$Al_2O_3$ and the number of intermediate modifications are sometimes quite contradictory [16-20]. Therefore, it was impossible to predict the phase composition of the detonation coating of aluminum oxide at first glance. In addition, the properties of detonation powder coatings, as well as coatings made by other methods, are significantly dependent on many factors, in particular on spraying modes. Therefore, the study of the structural features and properties of detonation coatings based on Al_2O_3 has the great interest.

Thus, the aim of our work is to study the effect of detonation spraying on the phase composition and mechanical properties of Al_2O_3 coatings.

Materials and methods of research

12X18H10T stainless steel was chosen as a substrate. The samples were sandblasted before coating. Corundum powders (α-Al_2O_3) were used to obtain coatings from aluminum oxide. The particle size of the powder is up to 22-45 μm. Detonation coatings were obtained on a computerized complex of new generation detonation spraying CCDS2000 (Computer Controlled Detonation

Spraying), [21-23].

In this work, we were obtained Al_2O_3 coatings by variation of the delay time between shots. Table 1 shows the technological parameters of the detonation spraying of Al_2O_3 coatings.

Table 1.
Technological parameters of detonation spraying of Al_2O_3 coatings.

Number of sample	Ratio O_2/C_2H_2	Barrel filling, %	Spraying distance, mm	Number of shots	Delay time between shots, s
1	1.856	63	250	20	1
2					0.75
3					0.5
4					0.25

Figure 1a presented a general view and a schematic diagram of the detonation spraying process. The channel inside the gun barrel is filled with gases using a high-precision gas distribution system, which is controlled by a computer. The process begins with filling the channel with carrier gas. A certain portion of the explosive mixture is supplied in such a way that a layered gas medium is formed after that, consisting of an explosive charge and a carrier gas. The powder is injected into the barrel (using a computer-controlled feeder) and forms a cloud by using a carrier gas stream. The substrate is placed at a certain distance from the exit from the trunk. The computer gives a signal to initiate detonation after powder part is injected. This is realized by using an electric spark. The duration of explosive combustion of a charge is about 1 ms. A detonation wave is formed in the explosive mixture, which in the carrier gas transforms into a shock wave. Detonation products (heated to 3500-4500 K) and carrier gas (heated by a shock wave to 1000-1500 K) move at a supersonic speed. The interaction time of gases with the sprayed particles is 2-5 ms. The particle velocity can reach 800 $m \cdot s^{-1}$.

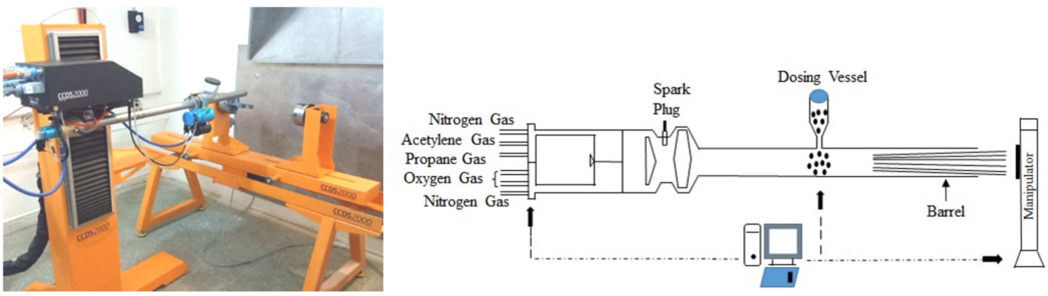

Figure 1. Computerized detonation complex CCDS200 (a) and its circuit scheme diagram (b).

The phase composition of the samples was studied by X-ray diffraction analysis on an X'PertPro diffractometer using CuKα-radiation. The Reference Intensity Ratio (RIR) method was used to determine the ratio of the main phases of the sample, which is intended for a quantitative phase analysis of mixtures when it is necessary to quickly evaluate the composition of the test sample with a low but acceptable accuracy. This method is based on reference intensity ratios (RIR values) and certain scale phase coefficients [24]. The measurement of hardness and elastic modulus was determined by the indentation method on the NanoScan - 4D compact nanotoughness tester in accordance with GOST R 8.748-2011 and ISO 14577 indentation with a load of 0.1 N.

Research results and its discussion

We have studied the nanohardness of coatings by the nanoindentation method. Figure 2 presents the comparative nanoindentation curves for coatings obtained under different conditions. It is seen that the penetration depth of the nanoindenter decreases with decreasing of delay time between shots.

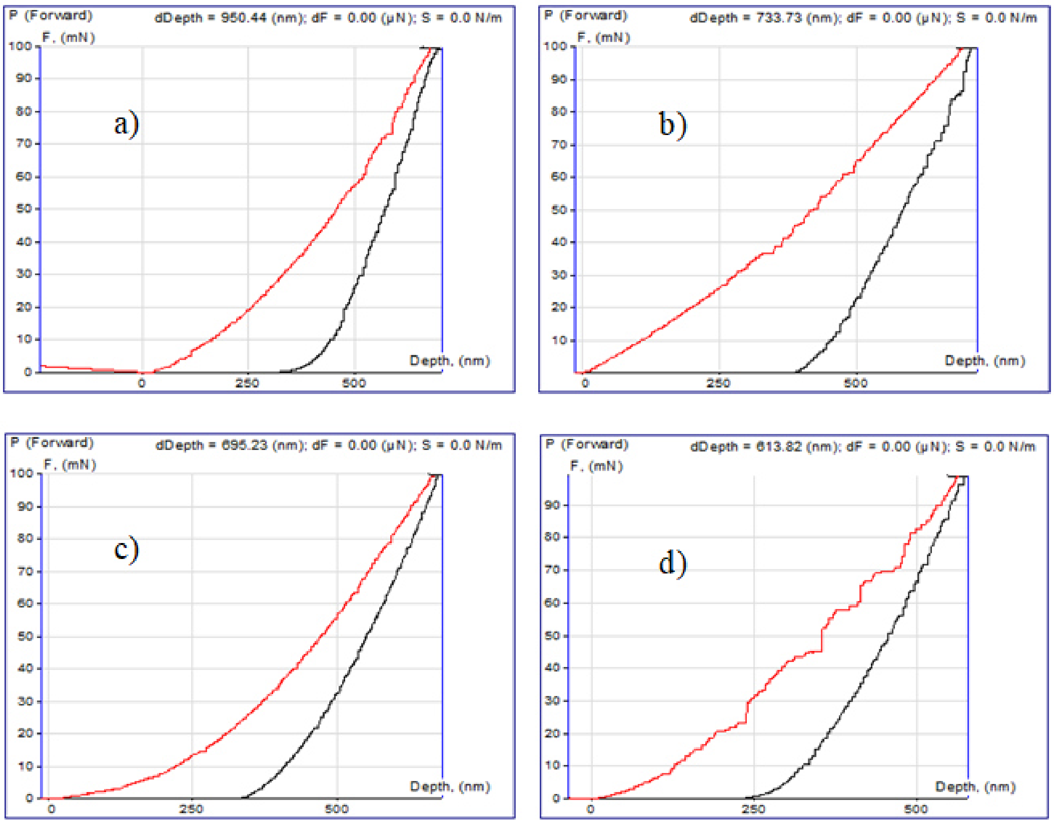

Figure 2. Comparative nanoindentation curves for Al_2O_3 coatings obtained under different spraying mode: delay time between shots - 1 s (a); 0.75 s (b); 0.5 s (c); 0.25 s (d).

Table 2 is shown the elasticity modules and nanohardness values calculated from the obtained curves. It can be seen that the hardness increases from 10.87 GPa to 16.33 GPa with decreasing in the delay time between shots. Also the elasticity modules of the coatings increase to 270.64 GPa. An increase in the elastic modulus indicates a decreasing in ductility and an increasing in the strength of the coatings. The reason for the increase in hardness is apparently due to the secondary thermal hardening of the coatings. Since, the speed and temperature of the particles do not change when the delay time between shots changes, but only the temperature of the applied coating layers changes during the deposition process. Thus, it is possible to obtain an Al_2O_3 coating with a hardness of 16.33 GPa and a Young's module of 270.64 GPa by reducing the delay time between detonation complex shots to the lowest possible value of CCDS2000.

Table 2.
Results of nanoindentation coatings of Al_2O_3 coatings.

Sample number	Nanoindentation, GPa	Young's module, GPa
1	10.87	207.70
2	11.03	159.97
3	11.72	206.48
4	16.33	270.64

Figure 3 presents the diffraction patterns of Al_2O_3 coatings obtained under different conditions. The results of X-ray diffraction analysis of the coatings showed that all coatings consist of γ-Al_2O_3 and α-Al_2O_3. The results of semi-quantitative analysis showed that despite of the fact that the initial powder was from α-Al_2O_3 and the coatings contained 65-75 % of the γ-Al_2O_3 phase. This is explained by the fact that nonequilibrium recrystallization of α-Al_2O_3 to γ-Al_2O_3 occurs under conditions of shock wave and rapid cooling during coating formation during the formation of coatings.

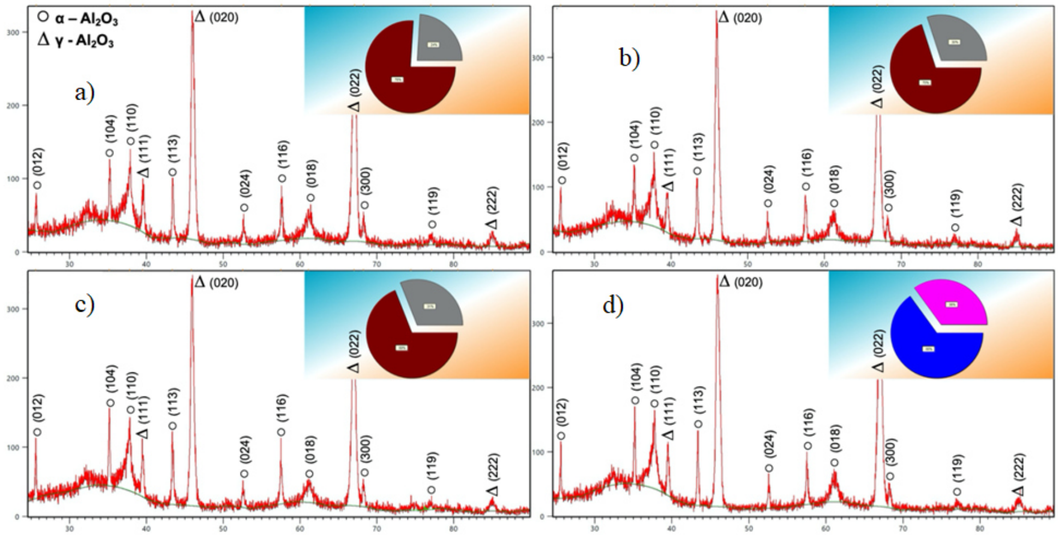

Figure 3. Diffractograms of Al_2O_3 coatings obtained under different spraying modes: delay time between shots - 1 s (a); 0.75 s (b); 0.5 s (c); 0.25 s (d).

The diffraction pattern shows that the volume fraction of these phases change depending on the spraying mode. The fraction of the α-Al_2O_3 phase increases with a decrease in the delay time between shots. This is due to the secondary recrystallization of $\gamma \rightarrow \alpha$, which occurs due to the heating of particles during the formation of coatings. It is known that recrystallization of γ-Al_2O_3 into α-Al_2O_3 by passing the intermediate phases begins at temperatures of $\approx 1100\,^{\circ}C$. Whereas in our case, reducing the delay time between shots to 0.25 s leads to an increase in the temperature of the applied coating layer above $1100\,^{\circ}C$.

Thus, reducing the delay time between shots of the detonation spraying method can be used as an additional energy source for heating the applied coating layer. Reducing the delay time between shots lead to the fact that the temperature in single spots of the coating will consistently increase when they are superimposed on each other. Obviously, the thermal effect of the spray spots will be noticeable in the order of a few milliseconds if the delay between the spraying of single spots is the minimum possible.

On the basis of X-ray diffraction analysis, it can be argued that the main reason of increasing in hardness with a decrease in the delay time between shots to 0.25 s is connected with increasing in the volume fraction of the α-Al_2O_3 phase. Modifications of α and γ phases has different values of physical-mechanical properties. α-Al_2O_3 modification have higher hardness and wear resistance, while γ-Al_2O_3 is relatively more elastic and provides higher adhesion to the substrate [25, 26].

Conclusion

An analysis of the results shows that Al_2O_3 coatings with high hardness can be obtained by reducing the delay time between shots. We obtained Al_2O_3 coatings with a hardness of 16.33 GPa and a Young's modulus of 270.64 GPa by reducing the delay time between shots to the minimum possible value for the CCDS2000 detonation complex. It is established that the main reason for the increase in hardness with a decrease in the delay time between shots to 0.25 s is associated with an increase in the volume fraction of the α-Al_2O_3 phase. X-ray diffraction studies presented that the highest content of the a phase is achieved when coatings are formed with a delay between shots of the order of 0.25 s. The increase in the volume fraction of the α-Al_2O_3 phase is caused by the secondary recrystallization $\gamma \rightarrow \alpha$, which occurs due to the heating of particles during coating formation, i.e. due to an increase in temperature above 1100°C in single spots of the coating when they are applied to each other. Thus, it is possible to control the phase composition of coatings based on Al_2O_3, and, accordingly, the properties of the coatings by changing the delay time between shots. The obtained results in the future make it possible to obtain gradient coatings that have high adhesive strength and high hardness of the coating surface by varying the delay time between shots during coating production. The preparation of such coatings is ensured by the fact that the surface layer of the coatings consists of a large amount of the α-Al_2O_3 phase, and the proportion of the γ-Al_2O_3 phase increases as it approaches the substrate. Thus, the more viscous γ-Al_2O_3 phase provides good adhesive strength of the coatings to the substrate, and the α-Al_2O_3 phase, which is found in large quantities in the coating surface, provides high hardness and wear resistance. In further works, we will present the results on obtaining gradient coatings based on these results.

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