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Influence of Detonation Spray Parameters on the Formation of Mechanical and Tribological Properties of Gradient Coatings based on Alumina

Using the detonation method, the shaft was filled with a gas mixture of C_2H_2/O_2 from 53 to 68 % and an alumina-based gradient coating was obtained on the surface of the substrate. The microstructure of the coatings was studied by scanning electron microscopic analysis. By increasing the proportion of α-Al_2O_3 phase towards the surface layer of the coating by 10–15 %, a coating layer with increased strength and wear resistance was received. By X-ray structural study, changes in the α-Al_2O_3 lattice were studied by reducing the amount of gas filling in the barrel and the firing time from 1 s to 0.25 s. By reducing the amount of gas in the shaft from 68 to 53 % and the firing time from 1 s to 0.25 s, a compatible gradient coating with improved mechanical properties was obtained, the maximum value of microhardness of the gradient coating was 23.73 GPa. The tribological properties of the coatings were studied and showed that the value of the coefficient of friction of the gradient coating is about 50 % lower than that of other coatings, i.e. wear-resistant.

Keywords: aluminum oxide, detonation spraying, gradient coating, phase, microstructure, friction coefficient, tribology, microhardness.

Introduction

The service life of metal parts can be significantly extended by coating them with special performance characteristics. Taking advantage of this situation, it is possible to make floor coatings from high-quality and inexpensive materials, as only a coating will improve the critical role of the part. Various engineering materials often suffer from mechanical loads, thermal or aggressive environments every day. Coating technologies are being developed, but the physical and mechanical properties are improved, i.e. the production of low-porosity, wear-resistant, adhesive-coated coatings is one of the unresolved issues. Everyone gets different coatings on their own, and most scientists are studying the properties of multi-layer or gradient coatings using several powders. Artechnology has its own advantages and disadvantages. Most surface coating technologies cannot spray several powders at once, i.e., to obtain a multi-layer or gradient coating. One of the most rapidly developing roofing technologies in recent years is the method of thermal spraying. Through this technology, it is possible to obtain high-quality, durable coatings [1–5].

Thermal spraying method includes detonation spraying (DS) and high-speed gas-flame spraying (HVOF), which allows getting a coating with excellent adhesive strength, low porosity, density and wear-resistance [6, 7]. Obtained by general thermal methods, alumina-based coatings consist mainly of the γ phase; γ phase has lower properties than α phase, has a low level of wear resistance and strength. The formation of a large number of γ phases is a common problem in all coatings obtained by air plasma, plasma, and gas-thermal spraying. In most cases, the raw powder consists only of α-Al_2O_3, the presence of α-Al_2O_3 after detonation spraying is due to the rapid crystallization of the molten volume of γ-Al_2O_3 because of insoluble or semi-soluble particles in the powder [8–10]. However, there are few studies on gradient coatings of aluminum oxide obtained by detonation spraying, which require further study. Therefore, we aim to obtain a gradient coating by changing the detonation parameters, studying the structural and phase state of aluminum oxide and changes in mechanical and tribological properties.

Experimental

Detonation coatings were obtained in the computerized complex of new generation detonation spraying CCDS2000 (Computer Controlled Detonation Spraying) [11, 12]. Figure 1 illustrates a general view and scheme of the detonation injection process. The shaft is filled with gases through a high-precision gas distribution system and controlled by a computer. The process begins by filling the shaft with carrier gas. Then a certain part of the explosive mixture is transferred in such a way that a layered gas medium is formed, consisting of a charge of explosive and carrier gas. Using a stream of carrier gas, it is poured into the explosion zone (using a computer-controlled feeder) and forms a cloud (fog). The mat is placed at a certain distance from the shaft. The computer beeps to start an explosion. This is done through an electric spark. The duration of explosive combustion of a charge is about 1 ms. A detonation wave is generated in the explosive mixture, which turns into a shock wave in the carrier gas. Detonation products (heated to 3500–4500 K) and carrier gas (heated to 1000–1500 k with shock waves) move at a speed higher than sound. The reaction time of gases with scattered particles is 2–5 ms. The velocity of the particles can reach 800–1200 ms^{-1} [13–15]. The method of detonation spraying was used to obtain Al_2O_3 coating. The size of the aluminum powder for spraying was 34±6 μm. The injection shaft was placed on a detonation unit “CCDS-2000” with a diameter of 20 mm and a length of 800 mm. Stainless steel **12Ch18N10T** was chosen as the lining material. The chemical composition of steel corresponds to GOST 4986–79 [16].

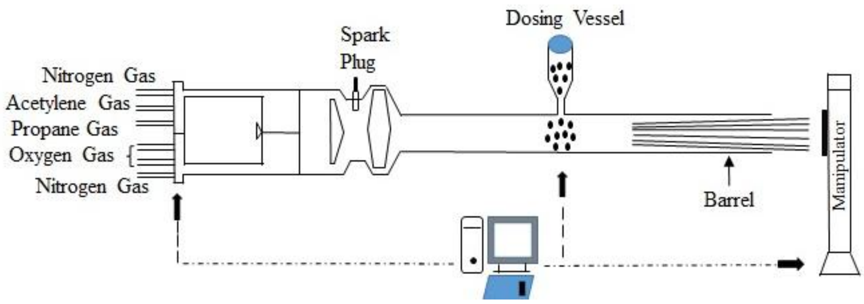


Figure 1. Schematic diagram of the computerized detonation complex CCDS200

The size of the substrate was 70x50x3 mm, the roughness of the initial surface was 0.080 μm. The surface of the substrate was sandblasted and chemically cleaned for 5–7 minutes. Sand with a grain size of about 40–60 microns was used to increase the average roughness of the dried substrate to 4.5 microns. In the production of a gradient coating based on alumina, the filling of the shaft with gas was between 53 %–68 %. The chemical composition of the substrate is shown in our previous study [17].

We obtained an Al_2O_3 coating for different volumes of barrel filling using a mixture of acetylene-oxygen as the fuel gas. As the proportion of explosive mixture increases (from 53 % to 68 % of the volume of the shaft), the temperature inside the shaft increases. In addition, changing the oxygen/fuel ratio from O_2 / C_2H_2 = 1.1 to O_2/C_2H_2 = 1.856 for low particle velocities can lead to an increase in temperature for the volume of explosive mixture. We chose O_2/C_2H_2 = 1.856 as the optimal ratio, which ensures complete melting of the powder material during injection. Table 1 shows the used process parameters.

Table 1

Technological parameters of Al_2O_3 coating

No.	O_2/C_2H_2	Barrel Filling Volume,%	Spray Distance, mm	Number of Shots
1	1.856	68, 63, 58, 53	250	20

We determined the phase composition of the sprayed coatings via the X-ray diffraction technique (XRD) applying an X'Pert PRO (Philips Corporation, Amsterdam, the Netherlands) diffractometer with Cu-Kα radiation (λ = 1.541 Å) at a voltage of 40 kV and a current of 30 mA. The diffractograms were decoded using the HighScore program. The measurements were carried out between 2°–90° with 0.02 step size and 0.5 s/step counting time. The surface roughness of the coatings was estimated according to GOST 2789–73 (ASTM D7127–05) using the Ra parameter by profilometer model 130 (JSC Plant PROTON, Moscow, Russia) [18]. We studied the mechanical properties of the coatings (microhardness) using a PMT–3M (LOMO, Saint Petersburg, Russia) microhardness tester. Microhardness was measured according to GOST 9450–76 (ASTM E384–11) [19] with a maximum load value equal to 3 N and a dwell time of 10 s. Tribological performances were evaluated in dry sliding tests performed on a high-temperature TRB3 (Anton Paar Srl, Peseux, Switzerland) using the standard ball-on-disc technique according to the ASTM G 133–95 and ASTM G99 standards [20, 21]. The sliding pair consisted of a stationary ball with a diameter of 6 mm and hardness 62±2 HRC, made of steel 100Cr6, pressed against a rotating disc made of steel **12Ch18N10T respectively** uncoated and spray-coated with 160–200 μm thick Al_2O_3 coating. The contact load amounted to 10 N and the rubbing speed to 0.2 m/s. The cycle time was 60 minutes. The tribological performance of the coatings was characterized by wear intensity and friction coefficient. To obtain results, we conducted the test on three samples from each variant. We used scanning electron microscopy (SEM) with backscattered electrons (BSE) at an accelerated voltage scanning electron microscope JSM-6390LV (Jeol, Tokyo, Japan) to study the cross-sectional morphology of the sample.

Results and Discussion

Figure 2 presents the microstructure of the cross-section for a gradient coating before and after annealing. The thickness of the coatings was 160–200 μm. The coating has a porous structure. The average pore size is 8–10 μm. Each sample was shot 5 times in each reduction, reducing the amount of gas in the barrel from 68 % to 63–58–53 %. The total number of shots was 20 times. A gradient coating was obtained by detonation spraying, reducing the volume of filling the shaft (gas) with gas (C_2H_2/O_2) from 68 % to 53 %. The roughness of the gradient coating is Ra = 0.269 μm. Based on the results of the study, it was shown that the change in the coating's roughness surface depends on the degree of gas filling of the shaft, as well as the proportion of α-Al_2O_3 and γ-Al_2O_3 phases in the coating.

Figure 3 shows the results of linear analysis of the lateral surface of the alumina-based gradient coating based on 68 %, 63 %, 58 %, 53 % of the shaft after gasification. Prior to the study of the coating, the surface of the coating was coated with carbon in a universal vacuum post, because it is difficult to see the surface morphology, as the aluminum oxide powder forms a ceramic when coated, so it was coated with a special carbon. Considering our study [17], the following gradient coating layer was obtained. On the surface of the substrate, we applied the first layer every 1 s with 68 % filling, and the second 5 layers every 0.75 seconds with 63 % filling, the third 5 layers every 0.5 seconds with 58 % filling and the fourth 5 layers every 0.25 seconds with the last 53 % filling.

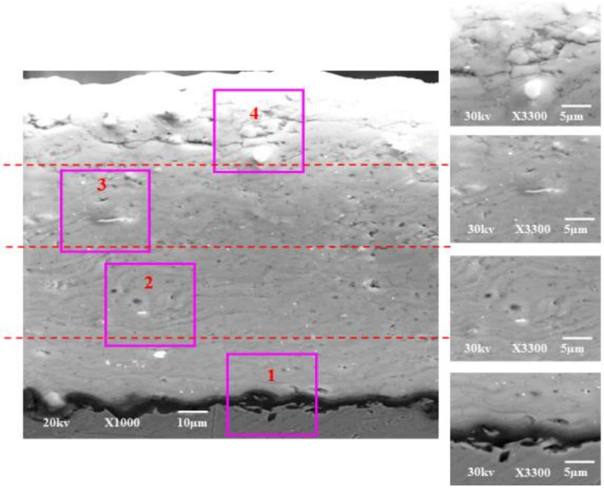
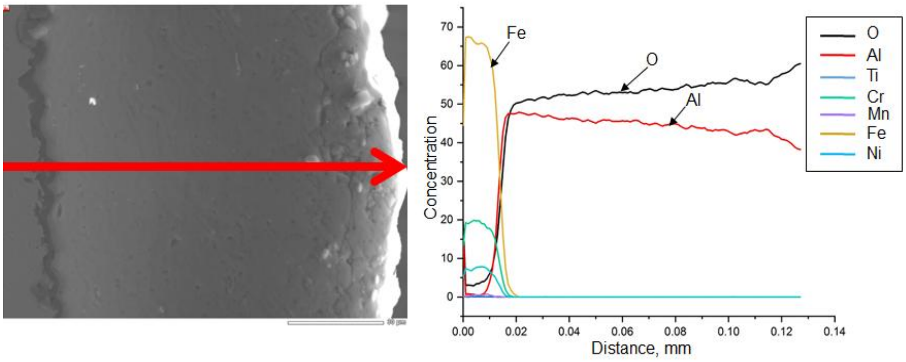


Figure 2. Microstructure of the cross-section of a gradient coating based on Al2O3:
68 % (1), 63 % (2), 58 % (3), and 53 % (4)

Figure 3a demonstrates that, in the area where the substrate is on the left side, the element Fe has more and less Ni and Cr elements. Towards the surface, one can see that the atoms of Al and O gradually increase, as shown in the right part of Figure 3a. The same process is noted in the distribution of the cardiogram elements shown in Figure 3b.



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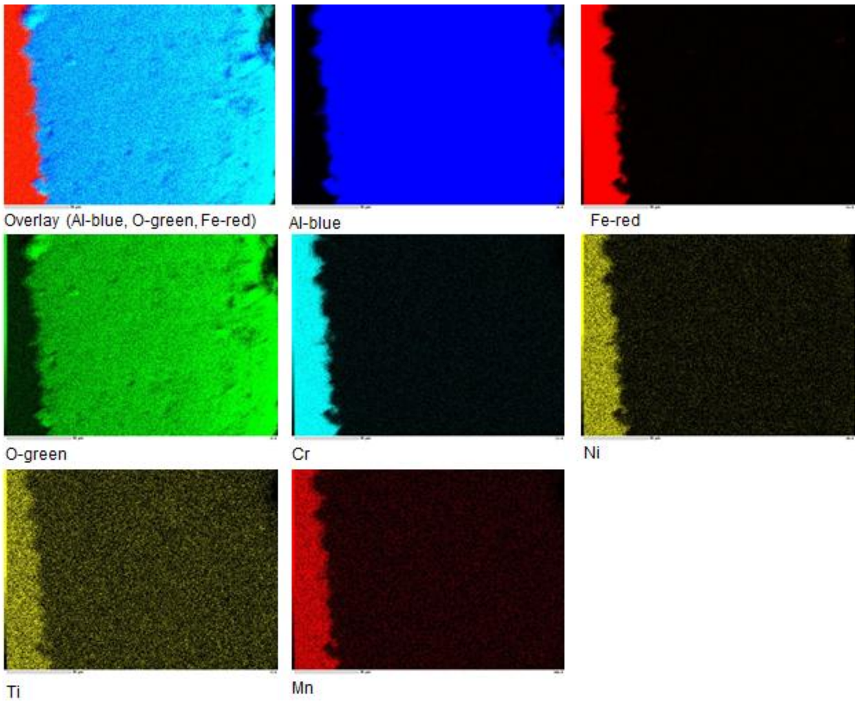

b

Figure 3. REM image taken from the cross-section of a gradient coating comprising aluminum oxide and the result of cardiogram analysis linear distribution of elements depending on the depth of the cross-section of the gradient coating Al_2O_3 (a); elemental distribution cardiogram (b)

Figure 4 shows the phase composition of the Al_2O_3 coating obtained using the given parameters. After detonation injection, alumina consisted of α-Al_2O_3 hexagonal (ICDD / JCPDS № 96–230–0376) and γ-Al_2O_3 cubic lattice (ICDD / JCPDS № 96–154–1583). Researchers [22, 23] state that Al_2O_3-based powder is capable of several modifications under the influence of high temperatures. Also, under the influence of heat, the composition of the Al_2O_3 coating changes into α, γ, δ, θ and different phases, in which the formation of α and γ phases predominates. These phases depend on the source material and the melting and cooling temperatures. Figure 4 comparatively shows the results of X-ray phase analysis of the shaft at 58 % gas filling and gradient coating. When the shaft is filled with gas up to 58 %, one can see that the proportion of the γ-Al_2O_3 phase in the alumina coating formed by the detonation explosion increases, while the proportion of the α-Al_2O_3 phase decreases (Figure 4a). We obtained the following gradient coating by regulating the phase change of the composition of aluminum oxide. First, by filling the shaft with 68 % gas on the surface of the substrate, we laid a layer with a larger share of the γ-Al_2O_3 phase. We continued to reduce the size of the γ-Al_2O_3 phase by slightly filling the next layer (ply) by 63 %, slightly increasing the proportion of the α-Al_2O_3 phase, filling the shaft by 58 % and continuing to reduce the γ-Al_2O_3 phase in the coating. We increased the proportion of α-Al_2O_3 phase in the coating by filling the shaft by 53 % to obtain the next layer, then continued the process. Our goal is to obtain a gradient coating with a single powder (aluminum oxide) using a single dispenser, based on which a coating layer with improved mechanical and tribological properties, the γ-Al_2O_3 phase in the coating increases the plasticity and elasticity of the coating [19, 24].

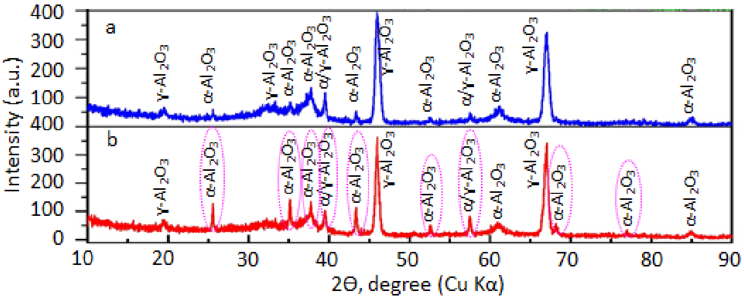

Figure 4. X-ray phase diffractogram of alumina coating obtained by filling the shaft with gas 58 % (a); 68 %, 63 %, 58 %, 53 % (b)

Figure 5 demonstrates a time graph of the coefficients of friction of the gradient coating relative to the substrate (Figure 5a) and the results of the study on the lost volume (Figure 5b). In [17], we stated that the coefficients of friction of coatings obtained during gas filling to 53–68 % were studied separately and μ = 0.52–0.59. The coefficient of friction of the pavement material was μ = 0.6–0.8, the value of the coefficient of friction of the gradient coating was μ = 0.027–0.33, i.e., the value of the coefficient of friction of the gradient coating, the wear resistance increased by 50 % compared to the pavement material. The value of the wear volume was 0.085 mm3 for the floor material, and 0.0165 mm3 for the gradient coating, i.e., it is 5 times less worn. The proposed gradient coating gives these steel products high physical, mechanical, and operational properties. In our opinion, the increase in hardness and wear resistance can be attributed to the increase in the proportion of α-Al_2O_3 phase in the coating. The α-Al_2O_3 phase has several features, including low density, relatively high melting point, excellent corrosion and wear resistance, high temperature resistance [24]. As can be seen, the wear resistance of the gradient coating was obtained by increasing the proportion of γ-Al_2O_3 phase in the substrate's vicinity and by increasing the amount of α-Al_2O_3 phase in the vicinity by reducing the degree of shaft filling from 68 to 53 % towards the surface layer. X-ray phase analysis explained this by an increase in the intensity of the diffractogram lines of the α-Al_2O_3 phase (Figure 4b).

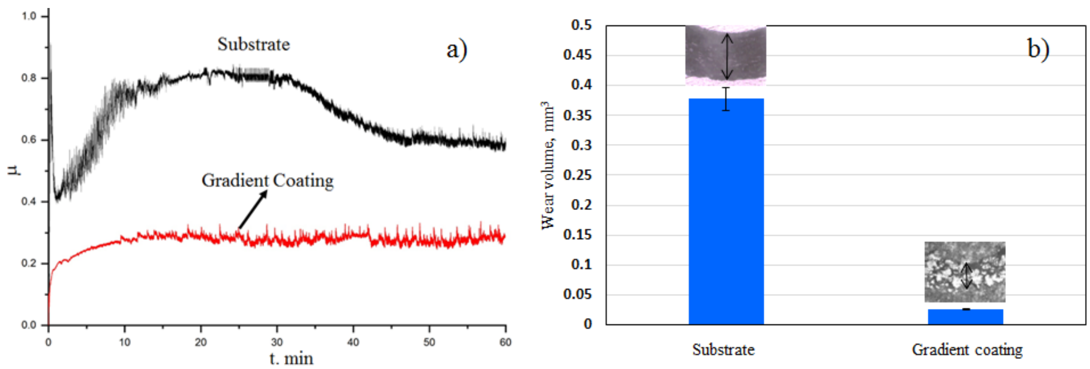

Figure 5. Results of tribological testing of Al_2O_3 coatings according to the scheme "ball-disk" a) change in the coefficient of friction; b) volumetric wear

In [17], the microhardness of the alumina coating obtained by filling the shaft with gas to 53–68 % was shown in between 16.31–20.56 GPa, the highest value of microhardness was observed at 53 % filling. The maximum value of the microhardness of the gradient coating obtained by changing the firing time with the degree of gas filling was 23.73 GPa, i.e. the value of the microhardness increased by about 15 % compared to the filling of 53–68 %. It was found that the microhardness increases when the barrel is filled with gas by 53 % and the firing delay is 0.25 s. In addition, the abrasion wear resistance of the gradient coating was compared with that of the substrate and standard steel 45, the abrasion resistance coefficient was 1 for standard material steel 45, 0.968 for substrate material, and 6.921 for Al_2O_3-based gradient coating. It was proved that the value of the coefficient of abrasive wear of the gradient coating obtained by us is 6–7 times higher than that of standard and lining material, i.e., wear-resistant.

Depending on the amount of gas filling of the shaft, the alumina-based coatings and gradient coatings were kept in a muffle furnace at 600 °C for 100 hours. Every 10 hours we turned off the oven, cooled it with the oven, took it out and measured its mass and microhardness, thus plotting the time dependence of the heat resistance coefficient as shown in Figure 6. The coefficients of wear resistance gradually decrease after 50 hours, when filling the shaft by 58, 63, 68 %, the gradient coating has little change compared to others. From this, one can see that the quality of the gradient coating obtained by the method developed by us is good, based on the characteristics of the mechanical, tribological, and structural-phase properties of the gradient coating studied above. We consider that the reason for this is the gradual decrease in the proportion of the γ-Al_2O_3 phase to the surface with a decrease in the delay time with the amount of gas filling the shaft, and the formation of a closely spaced gradient coating under the influence of an increase in the α-Al_2O_3 phase.

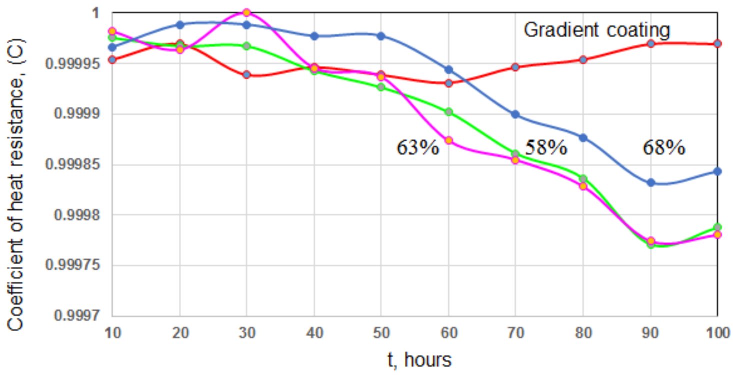

Figure 6. Graph of time dependence of Al2O3 gradient heat-resistance coefficient

Conclusions

1. A harmonious mode of obtaining a strong and dense coating layer was determined by reducing the percentage of gas filling of the detonation shaft from 68 to 53 % and regulating the formation of α-Al2O3 and γ-Al2O3 lattices of alumina-based coatings.
2. The microhardness of the alumina coating showed that the maximum value of the microhardness of the gradient coating obtained by reducing the amount of gas in the shaft from 68 to 53 % and the firing time from 1 s to 0.25 s was 23.73 GPa. The reason is the effect of increasing the volume fraction of the α-Al2O3 phase towards the surface.
3. In a tribological study of a gradient coating consisting of alumina obtained by the method developed by us, the value of the coefficient of friction was about 50 % less than that of the floor coating and other conventional coatings. The coefficient of abrasion wear resistance was 6–7 times higher than that of the lining material and steel 45, which indicates that the obtained gradient coating has a high wear resistance.
4. The value of the heat resistance coefficient of the gradient coating obtained by reducing the percentage of gas filling of the shaft from 68 to 53 % is uniform compared to other non-gradient coatings. that is, the α phase has higher physical and mechanical properties than the γ phase, i.e., it has a positive effect on temperature resistance.

Acknowledgements

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Детонациялық тозаңдау параметрлерін өзгерте отырып, алюминий оксиді негізіндегі градиентті жабынды алу және оның механикалық, трибологиялық қасиеттеріне әсерін зерттеу

Детонациялық әдісті қолданып, оқпанды (ствол) 53-тен 68 %-ға дейін C_2H_2/O_2 газ қоспасымен толтыра отырып, төсеніштің (подложка) бетіне алюминий оксиді негізіндегі градиентті жабын алынды. Алынған жабындардың микроқұрылымы растрлық электронды микроскопиялық талдау көмегімен зерттелді. Жабынның беткі қабатқа қарай α-Al_2O_3 фазаның үлесін 10–15 %-ға арттыру арқылы беріктігі, тозуға тұрақтылығы жоғарылаған жабын қабаты алынды. Рентгенқұрылымдық зерттеу арқылы, оқпанды газға толтыру мөлшері мен ату кезіндегі кідіру уақытын 1с-тан 0.25 с-қа азайту кезіндегі α-Al_2O_3 торларының өзгерісі зерттелді. Оқпандағы газдың мөлшерін 68-ден 53 %-ға және ату кезіндегі кідіру уақытын 1 с-тан 0.25 с-қа азайту арқылы механикалық қасиеті жақсартылған үйлесімді градиентті жабын қабаты алынды, алынған градиентті жабынның микроқаттылықтың ең жоғары мәні 23.73 ГПа-ды көрсетті. Алынған жабындардың трибологиялық қасиеті зерттеліп, градиентті жабынның үйкеліс коэффициентінің мәні басқа жабындармен салыстырып қарағанда 50 %-дай төмен, яғни тозуға берік екенін көрсетті.

Кілт сөздер: алюминий оксиді, детонациялық тозаңдау, градиентті жабын, фаза, микроқұрылым, үйкеліс коэффициенті, трибология, микроқаттылық.

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Получение градиентного покрытия на основе оксида алюминия путем изменения параметров детонационного напыления и изучение его влияния на механические и трибологические свойства

Детонационным методом ствол был заполнен газовой смесью C_2H_2/O_2 от 53 до 68 %, и на поверхности подложки было получено градиентное покрытие на основе оксида алюминия. Микроструктура полученных покрытий была исследована с помощью растрового электронно-микроскопического анализа. За счет увеличения доли фазы α-Al_2O_3 по отношению к поверхностному слою покрытия на 10–15 % был получен слой покрытия с повышенной прочностью и износостойкостью. Путем рентгеноструктурного исследования были изучены изменения решетки α-Al_2O_3 за счет уменьшения количества газа, заполняющего ствол, и времени задержки стрельбы от 1 до 0,25 с. Из-за сокращения количества газа в стволе с 68 до 53 % и времени задержки стрельбы от 1 с до 0,25 с было получено совместимое градиентное покрытие с улучшенными механическими свойствами, максимальное значение микротвердости полученного градиентного покрытия составило 23,73 ГПа. Были изучены трибологические свойства полученных покрытий и значение коэффициента трения градиентного покрытия примерно на 50 % ниже, то есть они показывали прочность к износу, по сравнению с другими покрытиями.

Ключевые слова: оксид алюминия, детонационное напыление, градиентное покрытие, фаза, микроструктура, коэффициент трения, трибология, микротвердость.

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