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Influence of Detonation-Spraying Parameters on the Phase Composition and Tribological Properties of Al_2O_3 Coatings

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Abstract: Al_2O_3 coatings were applied on the surface of 12Ch18N10T steel by the detonation method at different degrees of filling of the detonation gun. The aim was to study the influence of technological parameters on the formation of the coating's structure, phase composition and tribological characteristics. The degree of filling the gun with a gas mixture (C_2H_2/O_2) varied from 53% to 68%. X-ray diffraction study showed that the content of α-Al_2O_3 increases depending on the degree of filling. The results showed that the hardness increases with an increase in the α-Al_2O_3 phase. When the gun is 53% filled with gas, the Al_2O_3-based coating has the hardness of 20.56 GPa compared to 58%, 63% and 68% fillings. Tribology tests have shown that the wear rate and friction coefficient of the coating is highly dependent on the degree of filling of the gun.

Keywords: detonation spray; coatings; aluminum oxide; microstructure; phase; micro-hardness; friction coefficient

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1. Introduction

Engineering materials often undergo to the mechanical stress, heat radiation and/or corrosive environments. Taking this into account, not only new materials but the technologies have also been developed improving the performance of existing materials. The service life of machine parts and mechanisms can be extended by applying protective coatings or modifying the surface layer. One of the promising technologies is thermal spraying. Gas-thermal methods made it possible to obtain high quality, durable coatings [1–7]. For example, detonation spraying (DS) and high velocity oxygen-fuel (HVOF) spraying provide excellent adhesion strength, low porosity and a tough, dense and wear-resistant coating [8–10]. In addition, detonation spraying has the highest adhesion strength with the base material and the lowest porosity of the coating in comparison with other methods of gas-thermal spraying [11,12]. In this case, the quality of detonation coatings depends on the surface roughness of the substrate material and the chemical composition of the powder, the size of the granules, the ratio of gases and impurities [13]. With detonation spraying, coatings can be obtained from any materials, refractory compounds, oxides, etc. Al_2O_3, WC–Co, NiCrBSi and Cr_3C_2–NiCr are used to obtain wear-resistant coatings in order to restore parts.

Al_2O_3-based coatings are widely used to protect machine parts, boiler components, chemical and medical equipment, aircraft engine components, onshore and offshore turbines, etc. The final results of the research can be used in these areas. The powder of α-Al_2O_3 is usually used for detonation coatings. However, the coatings obtained by the detonation method mainly consist of the γ-Al_2O_3 phase. The γ-Al_2O_3 is formed because of rapid crystallization of the molten sample [14]. The content of α-Al_2O_3 in coatings after detonation spraying depends on the presence of insoluble or semi-soluble particles in the powder. Therefore, the structural-phase states of coatings based on aluminum oxide strongly depend on the technological mode of detonation spraying [15,16].

The aim of this research was to study the effect of filling degree of a detonation gun on the phase composition and tribological properties of detonation coatings of aluminum oxide.

2. Materials and Methods

Detonation coatings were obtained using the detonation spraying complex CCDS2000 (LIH SB RAS, Novosibirsk, Russia) [17,18]. Figure 1 shows a general view and diagram of the detonation injection process. The gun is filled with gases using a high-precision gas distribution system and is controlled by a computer. The process begins by filling the gun with carrier gas. Then a certain part of the explosive mixture is feed to form a layered gaseous medium, consisting of an explosive charge and a carrier gas. The powder is poured into the explosion zone with the carrier gas flow and forms a cloud (fog). The spray gun is placed at a certain distance from the barrel and the computer beeps to initiate the explosion. This is done with an electric spark. The duration of the explosive combustion of the 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 by shock waves) move at a speed exceeding the speed of sound. The reaction time of gases with scattered particles is 2–5 ms. The particle velocity can reach 800–1200 ms^{-1} [19,20]. The consumption of working gases at the average frequency of shots of 4 Hz is: acetylene 4–7; propane butane mixture 2–3, 5; oxygen 10–12; nitrogen 10–15 m^3/h. Nitrogen was used as a carrier gas.

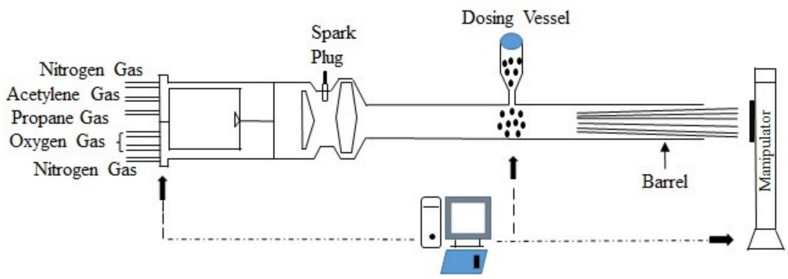


Figure 1. The diagram of CCDS2000 detonation complex.

The detonation spraying method was used to obtain Al_2O_3 coating. The size of the initial aluminum powder for spraying was 34 ± 6 µm. The injector was placed on a CCDS2000 detonation unit with a diameter of 20 mm and a length of 800 mm. Stainless 12Ch18N10T steel was selected as the substrate material. The chemical composition of the steel corresponds to GOST 4986–79 [21].

The dimensions of the steel samples were 75 mm × 50 mm × 5 mm and the surface roughness was R_a = 0.088 µm. The surface was cleaned with electrocorundum (Contracor Eco140S, Wuppertal, Germany) with a grain size of about 20–80 microns, and the roughness of the substrate was about 4–4.5 microns. The surface was chemically wiped for 5–7 minutes and the substrate was dried for 20–30 min, then coated with Al_2O_3 powder. Table 1 shows the chemical composition of 12Ch18N10T steel.

Table 1. Chemical composition of 12Ch18N10T steel (AISI 321).

C	Si	Mn	P	S	Cr	Mo	Ni	V	Ti	Cu	W	Fe
<0.12	<0.8	<2.0	<0.035	<0.02	17.0–19.0	<0.5	9.0–11.0	<0.2	<0.8	<0.4	<0.2	The rest

The thickness of the coatings was 180 μm. The average size of the formed coating in a single shot was 8–10 μm. Each sample was shot 20 times. The aluminum oxide coating was obtained by detonation spraying with different filling volumes (53%, 58%, 63%, 68%). Depending on the volume of gas filling the barrel in the detonation device, the degree of explosion will be different, i.e. the larger the gas filling volume, the higher the explosion power at the same gas ratios. The power of the explosion also depends on the type of gases used and their ratio [22,23]. Experimentally, we have found that when the filling volume is below 50%, the sprayed powder does not have a sufficient speed and temperature. During spraying, with a filling volume of 35%–45%, some of the powders are not applied and, hitting to the surface of the substrate, are pushed back. In order for spraying to occur, the powders must partially or completely melt and have a fixed speed depending on the size of the powder. Taking into account the above and the technical characteristics of the detonation installation (maximum filling volume up to 70%), We carried out experiments with filling volumes of 53%, 58%, 63% and 68%. In [24] it is shown that a changing in the technological modes of spraying, in particular, the frequency of spraying, leads to a changing in the phase composition, ie. changes in the content of α-Al_2O_3 and γ-Al_2O_3. Such changes make it possible to obtain gradient coatings, where the content of α-Al_2O_3 and γ-Al_2O_3 is adjusted in depth. Therefore, in this work, we studied the effect of the filling volume on the structure-phase states and tribological properties of coatings. Research data in the future allows to obtain gradient coatings by changing the filling volume during spraying.

Acetylene-oxygen mixture was used as a combustion gas. We chose O_2/C_2H_2 = 1.856 as the optimal ratio to ensure complete melting of the powder. Table 2 shows the process parameters.

Table 2. Technological parameters of Al_2O_3 coating.

№	O_2/C_2H_2	Barrel Filling,%	Spraying Distance, mm	Number of Shots
1	1.856	53	250	20
2	1.856	58	250	20
3	1.856	63	250	20
4	1.856	68	250	20

The distance between the barrel and the substrate was 250 mm. The surface of the substrate was subjected to sandblasting and dry cleaning for 5–7 min. The sand with a grain size of about 40–60 μm was used to increase the average roughness of the dried sample to 4.5 μm.

The phase composition of the coatings was determined by X-ray diffraction (XRD) on an X'Pert PRO diffractometer (Philips Corporation, Amsterdam, the Netherlands) with Cu-Kα radiation (λ = 1.541 Å) at a voltage of 40 kV and a current of 30 mA. Diffraction patterns were decoded using the "High Score" software; measurements were carried out in the 2θ range, equal to 10°–90°, with a step of 0.02 and a counting time of 0.5 s/step. The surface roughness was evaluated in accordance with GOST 2789–73 (ASTM D7127–05) [25] by the R_a parameter using a profilometer model 130 (JSC Plant PROTON, Moscow, Russia). The surface of the coatings was examined at optical magnification of ×20 using Altami MET 5S metallographic microscope (Altami LLC, St. Petersburg, Russia). The mechanical properties of the coatings (micro-hardness) were studied using PMT-3M micro-hardness tester (LOMO, St. Petersburg, Russia). Micro-hardness was measured according to GOST 9450–76 (ASTM E384–11) [26] at a maximum load of 3 N and a holding time of 10 s.

Tribological properties were assessed in dry slip tests using TRB-3 high temperature tribometer (Anton Paar Srl, Peseux, Switzerland) by a standard ball-disc technique in accordance with ASTM G 133–95 and ASTM G99 [27,28]. The sliding pair was a stationary ball with a diameter of 6 mm and a hardness of 62 ± 2 HRC, made of 100Cr6 steel. Pressed against a rotating disk made of 12Ch18N10T steel, respectively, without a coating and with a sprayed coating of Al_2O_3. The contact load was 10 N and a sliding speed was 0.2 m/s. The cycle time was 60 min. The tribological properties of the coatings were characterized by the wear rate and the coefficient of friction. To obtain reliable research results and to discuss the results, a test was carried out on three samples from each batch.

3. Results and Discussions

Figure 2 shows the structure of the substrate surface and Al_2O_3 powder after detonation injection using an optical microscope. Figure 3 shows the microstructure of the surface layer of the Al_2O_3 coating by scanning electron microscopy. According to the Figure 2, we see that the granules in the surface layer of steel 12Ch18N10T are large (coarse), and the surface layer of the Al_2O_3 coating is small (fine grained).

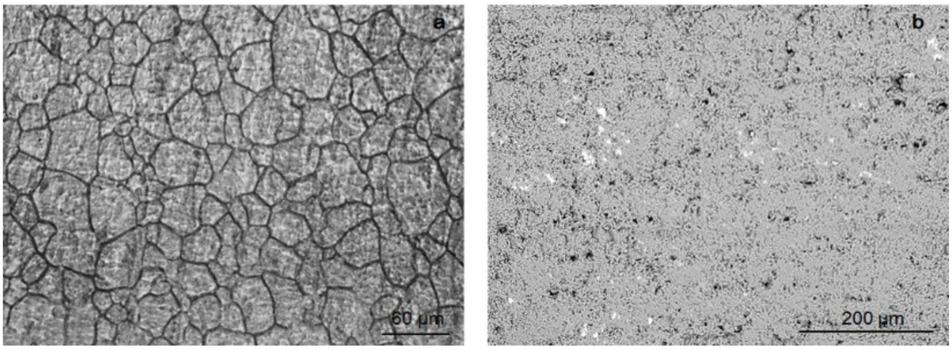


Figure 2. Surface structure of the substrate and structure of the surface after detonation spraying with Al_2O_3 powder: steel 12Ch18N10T (**a**), Al_2O_3 coating (**b**).

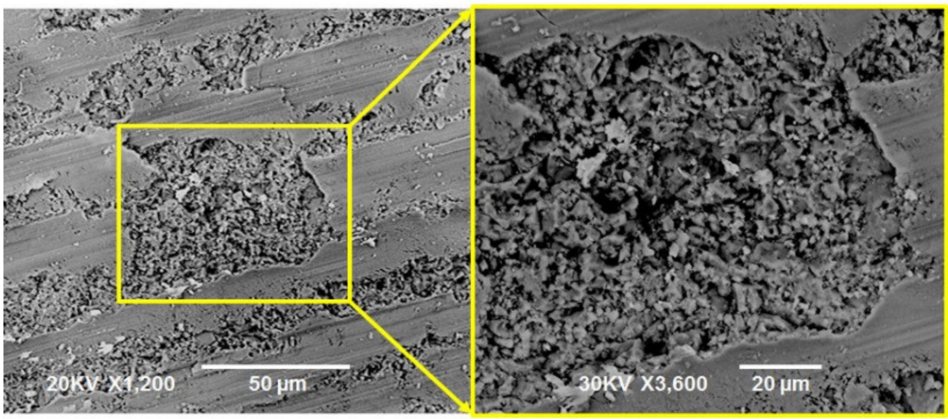


Figure 3. SEM image taken from the surface of the Al_2O_3 coating.

Figure 4 shows a graph of the microstructure and surface roughness, taken from the cross-section of the Al_2O_3 coating, depending on the volume of filling the barrel with gas 53%, 58%, 63% and 68%. R_a values of all coatings ranged from 0.439 to 1.442 µm. This shows that the degree of barrel filling does not greatly affect the roughness of Al_2O_3 coatings.

Figure 5 shows SEM image taken from the cross section of the Al_2O_3 coating when 53% of the barrel is filled with gas, and Figure 6 shows the results of spectral microanalysis. According to Figure 6, the coating is dense and uniform, as well as the composition of the coating in the spectrum 001–005 consists of pure Al and O atoms, as well as in the spectrum 006 taken from the boundary of the substrate and the coating, it was observed that the substrate material contains Fe, Cr, Ni atoms and the coating contains Al and O atoms.

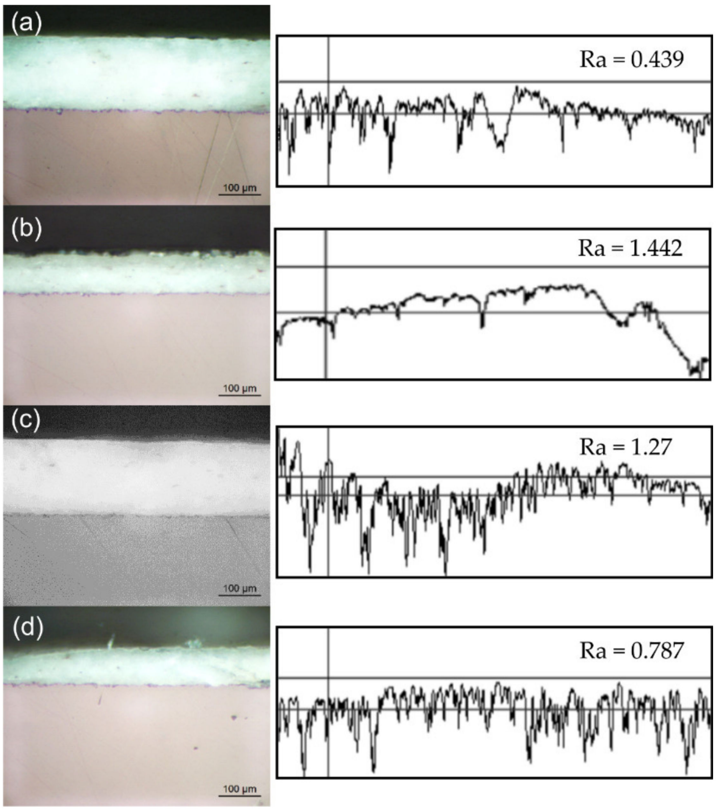


Figure 4. Depending on the volume of gas filling of the barrel the microstructure of the cross section of the Al_2O_3 coating and the surface roughness curve: 53% (**a**), 58% (**b**), 63% (**c**), 68% (**d**).

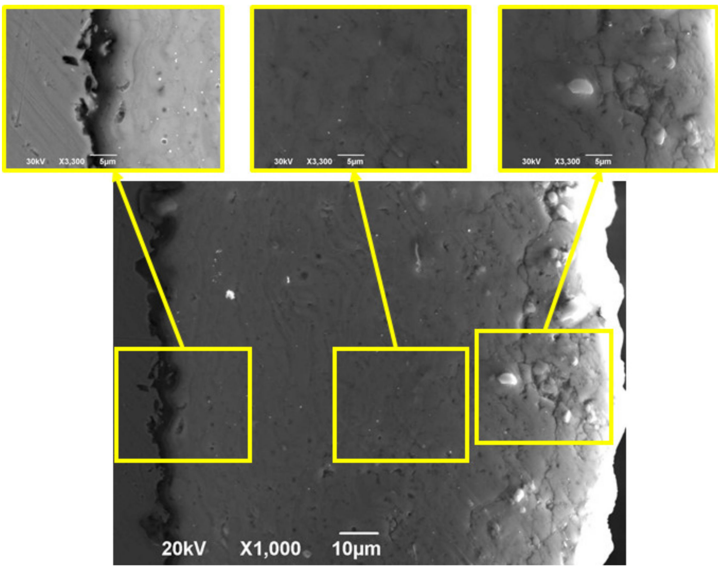
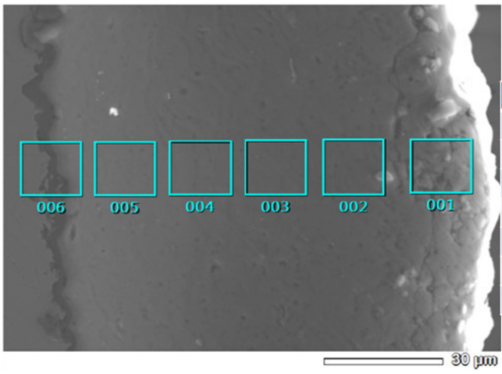


Figure 5. SEM image taken from the cross section of the Al_2O_3 coating when 53% of the barrel is filled with gas.



Atom, %						
spectrum	O	Al	Cr	Fe	Ni	Total
001	57.13	42.87				100.00
002	55.90	44.10				100.00
003	54.67	45.33				100.00
004	54.12	45.88				100.00
005	52.98	47.02				100.00
006	22.17	36.91	8.76	28.91	3.25	100.00

Figure 6. Spectral microanalysis of the cross section of the Al_2O_3 coating at 53% barrel gas filling.

Figure 7 shows the diffraction patterns of Al_2O_3 coatings obtained at 53%, 58%, 63%, and 68% barrel filling. The coatings after detonation spraying are composed of α-Al_2O_3 (ICDD/JCPDS No. 96–230–0376) and γ-Al_2O_3 (ICDD/JCPDS No. 96–154–1583). Corundum α-Al_2O_3 was used as a powder for spraying. The resulting coating mainly consists of γ-Al_2O_3. This is due to the fact that the powder of Al_2O_3 can undergo several modifications under the influence of high temperatures [29,30].

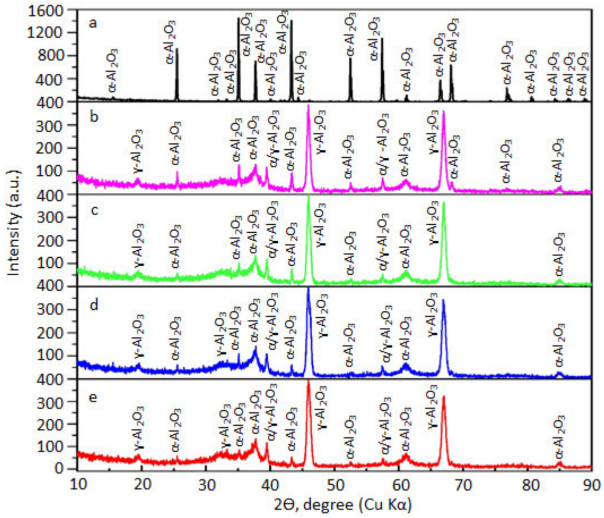


Figure 7. Diffraction pattern of aluminum oxide coating depending on the filling of the barrel: Al_2O_3 Powder (**a**), 53% (**b**), 58% (**c**), 63% (**d**), 68% (**e**).

Figure 7a–e shows the results of X-ray phase analysis of Al_2O_3 powder before detonation injection and when filling the barrel from 53% to 68%. Figure 7a shows that alumina oxide in powder form consists of a single α-Al_2O_3 phase. Figure 7 shows that with an increase in the filling of the barrel with gas from 53% to 68%, the content of γ-Al_2O_3 phase in the coating increases, while the content of α-Al_2O_3 phase decreases. In our opinion, it can be assumed that the γ-Al_2O_3 phase is formed when the barrel is filled by 68% due to an increase in the explosion temperature and the impact of a shock wave during flight along the barrel and subsequent spraying onto the surface. Since, it is known that when the temperature rises to 950–1250 °C, γ-Al_2O_3 turns into thermodynamically stable α-Al_2O_3. This transformation can proceed directly or through intermediate phases such as δ- and θ-Al_2O_3 [31,32].

Figure 8 shows the results of tribological tests of Al_2O_3 coatings by the ball-disk method. The wear resistance of the coatings was characterized by the volume of wear. Figure 8 shows that the samples have different values of the volume of wear depending on the degree of barrel filling. In this case, the wear gradually increases with an increase in the degree of filling. The coating obtained when the barrel is filled to 53% has a high wear resistance. Thus, based on X-ray diffraction analysis, it can be assumed that an increase in the volume fraction of α-Al_2O_3 phase in coatings leads to an increase in their wear resistance. This is because α-Al_2O_3 has a number of features, including low density, relatively high melting point, excellent corrosion and wear resistance, and high strength at high temperatures [33–35].

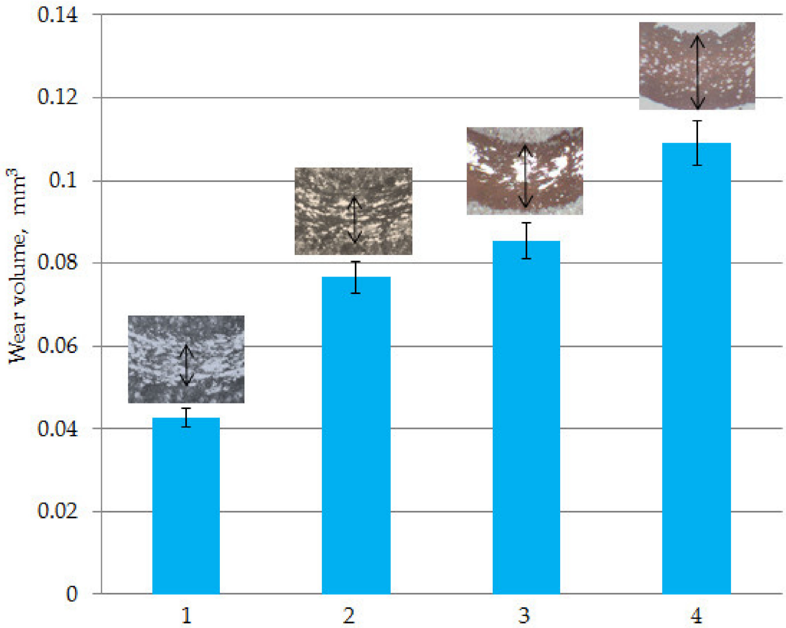


Figure 8. Results of tribological tests of Al_2O_3 coatings according to the "ball-disk" scheme: 53% (**1**), 58% (**2**), 63% (**3**), 68% (**4**).

Figure 9 shows the graphs of the friction coefficients of coatings obtained when filling the barrel by 53%, 58%, 63% and 68%. In [36] the tribological properties of the Al_2O_3 coating obtained by the plasma method using ZrO_2, Si_3N_4, Al_2O_3 and stainless steel balls (1Cr18Ni9Ti) with a diameter of 6 mm. It was noted that the coefficient of friction was $\mu \approx 0.55$–0.6 with ZrO_2, with Si_3N_4 $\mu \approx 0.5$–0.55, with Al_2O_3 ball $\mu \approx 0.6$–0.65, with stainless steel ball (1Cr18Ni9Ti) $\mu \approx 0.7$–0.8. In [37] a steel ball 100Cr6 was used to study the friction and wear resistance of ZrO_2–Al_2O_3-based coatings obtained by air-plasma injection. In our study, a steel ball 100Cr6 was used, respectively, the value of the coefficient of friction was in the range $\mu \approx 0.52$–0.59, the coefficient of friction was observed in the coating when filling the barrel with gas at least 53%. The coefficients of friction of all coatings obtained when filling the barrel by 53%, 58% and 68% have the same values and are $\mu = 0.52 \pm 0.01$. The coating obtained when filling the barrel by 63% has a high coefficient of friction $\mu = 0.59 \pm 0.01$ in comparison with other samples. Apparently, this is due to the roughness and porosity of the coatings.

Figure 10 shows the change in the micro-hardness of the coating. Modifications of α- and γ-phases have different values of physical and mechanical properties. Modifications of α-Al_2O_3 have high hardness and wear resistance, while γ-Al_2O_3 is relatively elastic and provides high adhesion to the substrate [38]. Analysis of the effect of phase composition on the physicochemical and mechanical characteristics of plasma detonation coatings made of aluminum oxide shows that an increase in the stable γ-phase, as well as other (metastable) phases reduces the hardness, wear resistance and corrosion resistance of the coating. In addition, the maximum hardness of the coating obtained by plasma detonation is 1.98 Gpa, Thus, the use of plasma-detonation technology for spraying coatings made of Al_2O_3 allows to obtain dense multi-phase coatings with good adhesion to the substrate (due to damping of low-temperature stable and metastable modifications) and high physical and mechanical properties (due to high α-phase composition) [13]. Based on these analyzes, based on the results of our study, we conclude that the mechanical and tribological properties increased due to the increase in α-Al_2O_3, and the adhesion strength may improve due to the γ-Al_2O_3 phase. In [28] reported that the hardness of the Al_2O_3 coating obtained increased from 10.87 to 16.33 GPa by reducing the delay time between shots during a detonation explosion from 1 to 0.25 s. In [17] reported that the hardness of the Al_2O_3 coating increases from 7.86 to 11.08 GPa as the thickness increases from 250 to 1100 μm. In [35] reported that the microhardness of ZrO_2–Al_2O_3-based coatings obtained by air-plasma injection was in the range of 8.06–9.11 GPa. The micro-hardness of the obtained coatings, depending on the filling with gas, was at 53%–20.6 ± 0.3 GPa, 58%–16.3 ± 0.15 GPa, 63%–17.9 ± 0.25 GPa, and at 68%–18.16 ± 0.27 GPa. At the same time, the highest value of micro-hardness was observed at 53% filling. An increase in the micro-hardness of coatings is associated with an increase in the α-Al_2O_3 phase. γ-Al_2O_3 has a lower hardness and mechanical resistance than α-Al_2O_3.

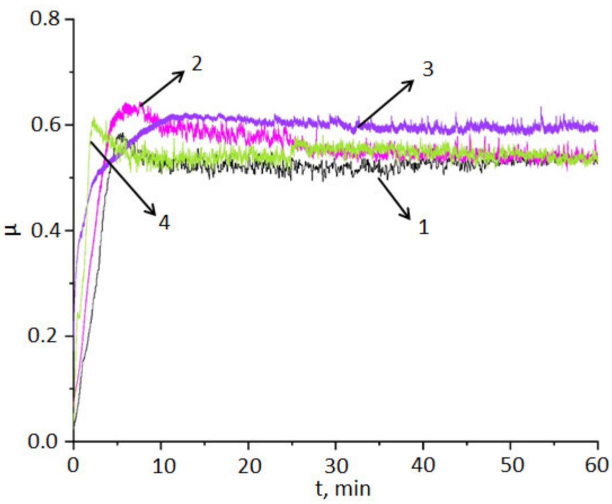


Figure 9. Results of tribological studies of Al_2O_3 coating depending on the filling of the barrel with gas: 53% (**1**), 58% (**2**), 63% (**3**), 68% (**4**).

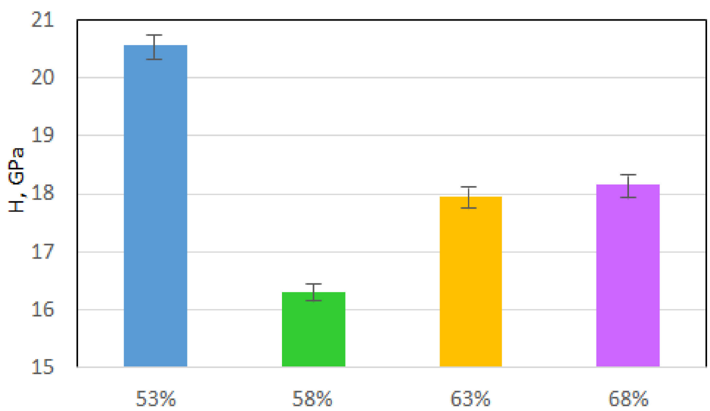


Figure 10. Changes in the micro-hardness of the surface layer of an aluminum oxide coating depending on the filling of the barrel with gas.

4. Conclusions

Thus, it can be concluded that the detonation spraying method can be used to obtain coatings based on Al_2O_3 with specified structure and properties. During detonation spraying of Al_2O_3 powder, the resulting coating consists of α-Al_2O_3 and γ-Al_2O_3, and their volume fraction strongly depends on the technological mode of spraying. It is clear that the amount of wear gradually decreases with increasing barrel filling. The coating obtained when the barrel is filled to 53% has a high wear resistance. Based on X-ray diffraction analysis, it was found that an increase in the volume fraction of α-Al_2O_3 phases in coatings leads to an increase in their wear resistance. It is necessary to increase the volume fraction of α-Al_2O_3 to ensure high hardness and wear resistance of the detonation Al_2O_3 coating. In further studies, it is planned to obtain a gradient coating based on Al_2O_3, in which the structure and properties gradually change with depth.

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