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Effect of powder morphology on the structure and properties of Al₂O₃ based coatings obtained by detonation spraying

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Abstract. Aluminum oxide (Al₂O₃) remains a crucial material in the field of engineering ceramics, particularly in industrial applications. Notably, its transitional phases, in addition to the α -phase, demonstrate good properties. In this article the structure and properties of coatings obtained by detonation spraying from aluminum oxide powder with different production methods (fragmentation and spherical particle shape) and crystalline modification (α -Al₂O₃ and γ -Al₂O₃) but with similar average particle size were studied. The goal of the study was to investigate the influence of the morphology and phase composition of the powder on such properties of the coatings as microstructure, porosity, roughness and hardness. The results of the study showed that agglomerated spherical powder leads to low porosity of the coatings, while crushed fragmentation particles provide dense layers. In addition, the coating obtained using fragmentation powder showed higher hardness, and the surface had twice the roughness, compared to the coating applied using spheroidal powder. These results confirm the effect of the morphology of the original powder on the final properties and efficiency of the coatings. The study showed that the detonation spraying method allows for the efficient application of coatings from two types of Al₂O₃ powders with different morphology (spherical and fragmentation) and phase composition (γ -Al₂O₃ and α -Al₂O₃) but with similar particle sizes.

Keywords: detonation spraying, aluminum oxide, powder morphology, ceramic coating, porosity, hardness

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1. INTRODUCTION

In current engineering practice, special attention is paid to increasing the durability and reliability of structural materials in various industries, including construction. One of the main directions in achieving these goals is the use of functional coatings that repeatedly improve the properties of structural materials. Modern technologies allow the formation of various types of coatings on the surfaces of different materials, which not only create a protective barrier from various external and environmental factors, but also provide high wear resistance, corrosion resistance and increased strength [1, 2].

Among the many available ceramic coatings, aluminum oxide (Al_2O_3) coatings are promising materials for use in conditions requiring high wear resistance and corrosion resistance [3, 4]. However, the physical and mechanical characteristics of these coatings depend on many factors, including the parameters of the application process, phase composition, size and morphology of the particles of the original raw materials. Even small changes in these factors can lead to significant differences in the structure and properties of the coatings. Recent studies have shown that coatings produced by the plasma spraying method from powders with different particle morphology, but with a uniform chemical composition and similar particle size distribution, can lead to significant structural differences in the coating [5]. In addition, the authors of [6] showed that the use of $\gamma\text{-Al}_2\text{O}_3$ based powder as a raw material for plasma spraying allows obtaining coatings with a predominant $\alpha\text{-Al}_2\text{O}_3$ phase. This shows that it is possible to control the phase composition of the coating by choosing the appropriate powder.

Other studies have shown that the particle size of the starting powder can affect the properties of the resulting coating. Han, B. et al. [7] used $\alpha\text{-Al}_2\text{O}_3$ powder with different particle sizes (45.8 μm and 22 μm) as a feedstock and found that the coating obtained using the larger powder size (45.8 μm) had higher porosity and bonded crack density compared to the coating obtained using the smaller powder size (22 μm). In addition, particle temperature measurements in spray systems showed that with increasing particle size, the proportion of partially melted particles increases, which leads to an increase in the content of the $\alpha\text{-Al}_2\text{O}_3$ phase in the coating [8]. Rakhadilov et al. [9] carried out a comparative analysis of the structure and properties of detonation coatings containing the main phases of $\gamma\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$. The results of the study showed that the $\gamma\text{-Al}_2\text{O}_3$ phase has higher adhesion strength compared to the $\alpha\text{-Al}_2\text{O}_3$ phase. In addition, the authors of [10] found that the improvement of the hardness and hardening synergy in Al_2O_3 based coatings can be achieved by creating an optimal combination of $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ phases. The $\alpha\text{-Al}_2\text{O}_3$ phase is known for its high hardness and wear resistance, while the $\gamma\text{-Al}_2\text{O}_3$ phase has high adhesion strength and hardening. Therefore, adjusting the α/γ phase ratios in Al_2O_3 coatings can lead to the creation of coatings with improved properties, such as increased hardness, wear resistance, and adhesion strength. These results highlight the importance of controlling the spraying process parameters and raw material properties to achieve optimal coating performance.

Among the many available coating application technologies, the detonation thermal spraying method based on a multi-chamber detonation accelerator (MCDA) is a promising method [11-15]. The design features of the MCDA allow for detonation combustion of mixtures with a low content of combustible components while maintaining the temperature of the combustion products. Detonation combustion of the gas mixture is initiated with a detonation frequency of 20 Hz and higher, which ensures a high coating application rate. Coatings obtained using the MCDA are characterized by low porosity (<1%), excellent adhesion and strength. In addition, the MCDA allows for the production of coatings up to 1000 μm thick at the industrial level, which opens up new opportunities for the production of products with improved properties [16, 17]. The goal of this study is to obtain a high-quality Al_2O_3 based coating with minimal porosity using the detonation spraying method implemented using a multi-chamber detonation accelerator. In addition, it is planned to study the effect of the morphology and phase composition of raw powders on the process of coating structure formation to optimize the properties of the resulting coatings.

2. METHODS AND MATERIALS

The Al₂O₃ ceramic coatings were obtained by the detonation spraying method using a multi-chamber detonation device (MCDD) [18] in accordance with the parameters presented in Table 1.

Table 1. Modes of Al₂O₃ Coating Deposition.

Parameters	α -Al ₂ O ₃ powder	γ -Al ₂ O ₃ powder
Consumption of combustible mixture components: [m ³ /hour]		
- air	0.14*/0.14**	1.41*/0.85**
- oxygen	4*/4**	3.13*/3.68**
- propane/butane (40/60 %)	0.75*/0.68**	0.63*/0.68**
carrier gas - air	0.9	
Powder feed rate [g/hour]	200	110
Spraying distance [mm]	50	
Barrel length [mm]	500	
Inner diameter of the nozzle [mm]	16	
Speed of movement along the sample [mm/min]	2000	
Number of spray passes	6	6

* – cylindrical combustion chamber, ** – annular combustion chamber.

Two Al₂O₃ powders with different morphology and phase composition were used for the study (Fig. 1). The first powder consisted of crushed fragmentation-shaped particles with the α -Al₂O₃ phase, the second one consisted of agglomerated and sintered spherical particles with the γ -Al₂O₃ phase. The particle size distribution histograms show similarity in the sizes of both fractions. The fragmentation powder had an average particle size of 11.83 μ m (Fig. 1a), and the spherical powder - 10.76 μ m (Fig. 1b). Both powders were applied to steel substrates measuring 40 × 40 × 4 mm. Before the deposition process, the substrates were abrasive blasted using dry silica abrasive with a grain size of 1 mm and then blown with high-pressure air. The surface and cross-sectional morphology of the Al₂O₃ coatings were characterized using scanning electron microscopy (TESCAN MIRA 3 LMU). The porosity of the coating was determined by image analysis using ImageJ software according to the ASTM standard ASTM E2109-01(2014) [19]. The porosity was estimated as the average of four measurements in micrographs randomly obtained on polished cross-sections of the coatings. A detailed procedure for porosity estimation is described in [20].

Phase analysis of the powders and coatings was performed using X-ray diffraction (XRD) in the range of 10–80° using Co K α radiation (ARL 9900 WorkStation). Three-dimensional surface topography was performed using the SIAMS-800 software and hardware complex (LLC «SIAMS», Yekaterinburg, Russia). The Al₂O₃ coating application modes are presented in Table 1.

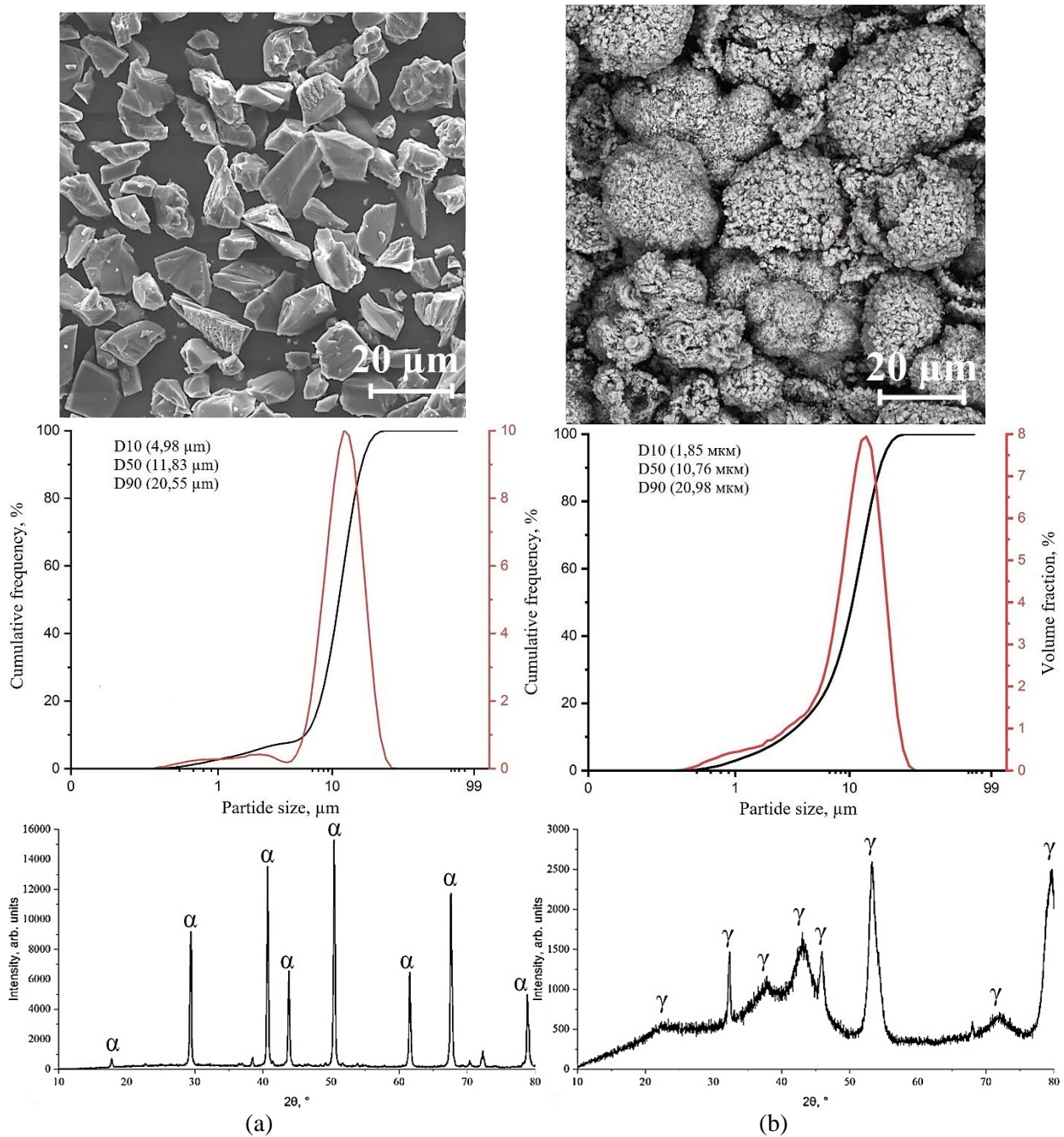


Fig. 1. Morphology, particle size distribution, and X-ray diffraction profile of powders: a) α - Al_2O_3 and b) γ - Al_2O_3 .

The coating hardness was measured for polished sections with a Vickers indenter under a load of 2.94 N (HV0.3) using a calibrated Nexus 4000 device. For each sample, 10 indentations were made in the upper, middle and lower parts of the coating section. The average hardness and its standard deviation were then calculated.

3. RESULTS AND DISCUSSION

The powder (material) utilization factor is an important indicator of the coating application process efficiency. Its increase can lead to an improvement in the quality and properties of the resulting coatings. When using optimal application modes, it is possible to achieve a high level of powder efficiency, which can lead to material savings and a reduction in the cost of obtaining coatings. During

the study, optimization regimes for applying Al_2O_3 coatings with different particle morphology and phase composition were selected. The results showed that when using optimal application regimes, the powder utilization factor reaches high values: 65% for $\alpha\text{-Al}_2\text{O}_3$ and 80% for $\gamma\text{-Al}_2\text{O}_3$.

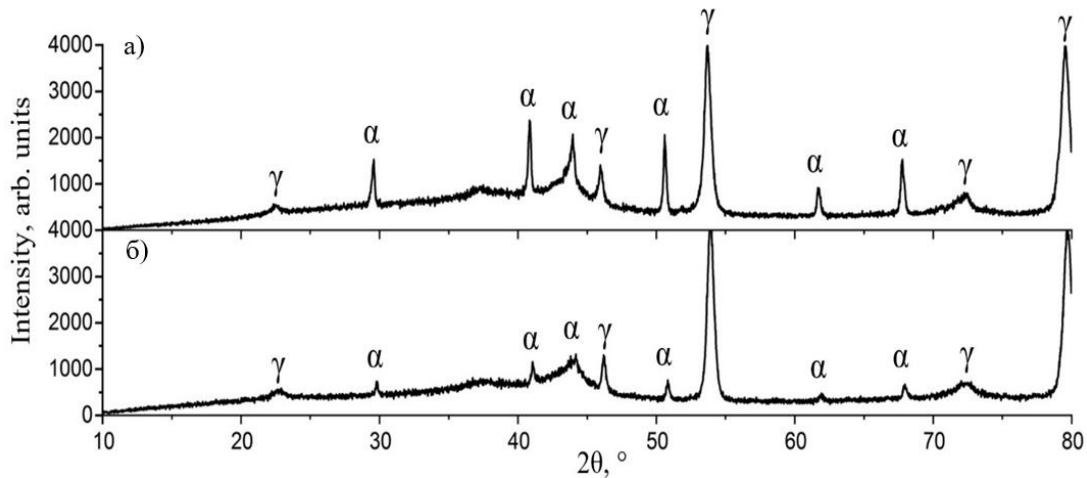


Fig. 2. X-ray diffraction profiles of Al_2O_3 coatings based on powders: a) $\alpha\text{-Al}_2\text{O}_3$ and b) $\gamma\text{-Al}_2\text{O}_3$.

Fig. 2 shows the diffraction profiles for the Al_2O_3 coatings obtained from $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ powders. Two main phases, $\alpha\text{-Al}_2\text{O}_3$ (ICDD/JCPDS No. 5-712) and $\gamma\text{-Al}_2\text{O}_3$ (ICDD/JCPDS No. 10-425), were revealed in the coatings formed by the detonation spraying method. Despite the use of different initial powders, the phase composition of the obtained coatings does not differ significantly. As a result of the impact of a shock wave and rapid cooling during the formation of the coating, none-equilibrium recrystallization occurs. Upon rapid cooling of molten Al_2O_3 particles, metastable $\gamma\text{-Al}_2\text{O}_3$ is formed instead of $\alpha\text{-Al}_2\text{O}_3$, since $\gamma\text{-Al}_2\text{O}_3$ has a lower interfacial energy between the crystal and liquid phases [21, 22]. Therefore, it can be assumed that during spraying using two powders with different morphology and phase ($\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$), the molten particles formed by rapid heating may have different degrees of heating at the time of deposition on the substrate surface. The particles obtained from the $\gamma\text{-Al}_2\text{O}_3$ powder may have a higher degree of heating, a longer cooling period and a longer time to hold the temperature ($\sim 1100\text{ }^\circ\text{C}$) of the phase transition compared to the particles obtained from the $\alpha\text{-Al}_2\text{O}_3$ powder. This may affect the phase composition of the coating. Thus, using the detonation spraying technology, it is possible to obtain coatings with more preferable structure and properties from the $\gamma\text{-Al}_2\text{O}_3$ powder.

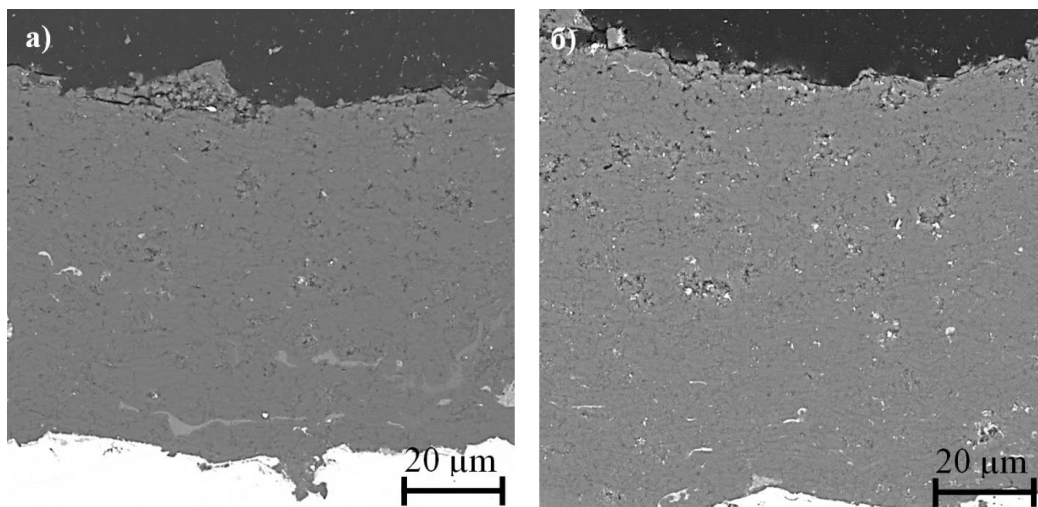


Fig. 3. Cross-sections of Al_2O_3 coatings based on powders: a) $\alpha\text{-Al}_2\text{O}_3$ and b) $\gamma\text{-Al}_2\text{O}_3$.

Fig. 3a and 3b demonstrate the microstructure of the cross-section of the coating. The average thickness of the Al_2O_3 coatings is $\sim 100 \mu\text{m}$. In the images, the black layer represents the epoxy resin used to fix the sample. The boundary between the coating and the substrate is clearly visible. No defects were found. The main mechanism of adhesion between the coating and the substrate is mechanical. Most of the Al_2O_3 component, regardless of the morphology and phase composition of the powder, melts well and has a flattened shape, i.e., a lamellar structure. The coating shown in Fig. 3a, formed from $\alpha\text{-Al}_2\text{O}_3$ powder with a fragmentary particle shape, is characterized by a dense structure free of micro-cracks and other defects. The number of pores in the cross-section of the coating is minimal, and the pores are almost round. The coating (Fig. 3b) obtained from $\gamma\text{-Al}_2\text{O}_3$ powder with an agglomerated spherical particle shape is characterized by a less dense and uniform microstructure. It contains pores and voids, which are associated with different degrees of fusion and distribution of powder particles during the spraying process. This difference in microstructure is due to the peculiarities of coating formation during detonation spraying, where the geometry of the particles and their interaction during the spraying process can affect the density and homogeneity of the resulting layer.

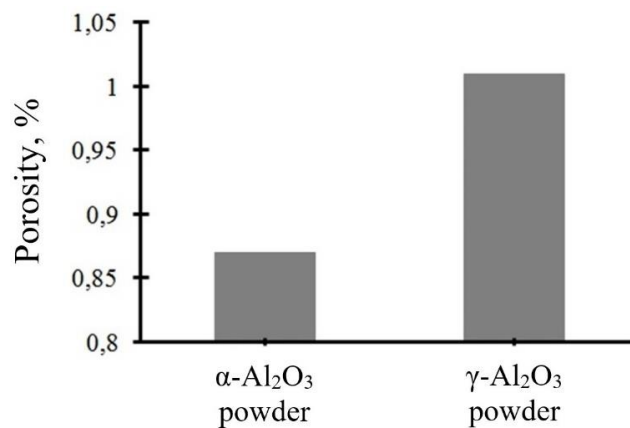


Fig. 4. Cross-sectional annotation of pores of Al_2O_3 coatings based on powders.

The porosity of the coatings was 0.87% for the coating obtained from the $\alpha\text{-Al}_2\text{O}_3$ powder with a fragmented particle shape, and 1.01% for the coating obtained from the $\gamma\text{-Al}_2\text{O}_3$ powder with an agglomerated spherical particle shape (Fig. 4). This is due to the fact that the agglomerated spherical powder contains more voids and pores in the particles than the fragmented powder. The analysis showed that the pores are randomly distributed in the coatings.

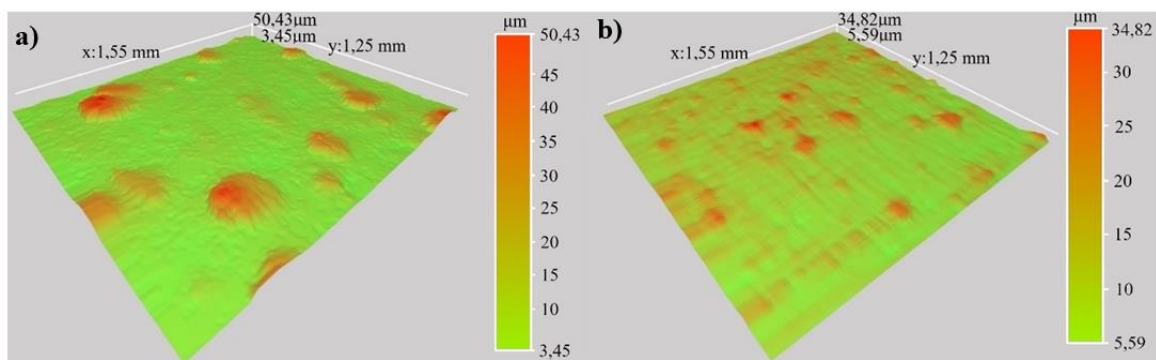


Fig. 5. Three-dimensional surface topography of Al_2O_3 coatings based on powders: a) $\alpha\text{-Al}_2\text{O}_3$ and b) $\gamma\text{-Al}_2\text{O}_3$.

Fig. 5 shows a quantitative assessment of the surface morphology of the Al_2O_3 coatings. Visual analysis of the image shows that both Al_2O_3 coatings have a rough surface, which can be characterized as "bumpy". These "bumps" are distributed randomly over the surface. For the coating obtained from the fragmented powder, the maximum height is $46.98\ \mu\text{m}$, while for the coating obtained from the spherical powder – $29.23\ \mu\text{m}$. Thus, the formation of the coating surface directly depends on the morphology of the original powder, fed with high kinetic energy and then subjected to impact and melting during the braking process.

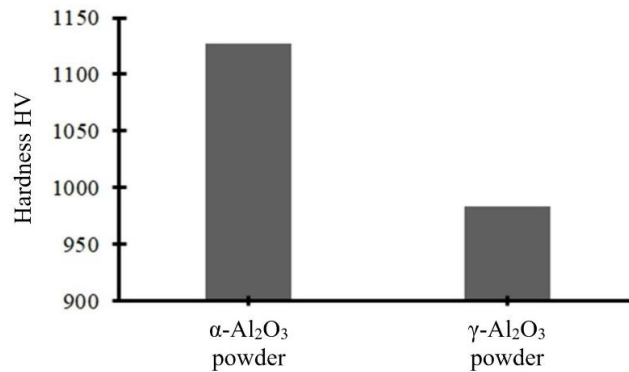


Fig. 6. Vickers hardness of Al_2O_3 based coatings.

The results of micro-hardness measurements performed using a Vickers hardness tester are shown in Fig. 6. The coating based on $\alpha\text{-Al}_2\text{O}_3$ powder has a maximum hardness of $1127\ \text{HV}_{0.3}$. Similarly, the maximum hardness of the coating based on $\gamma\text{-Al}_2\text{O}_3$ powder is $983\ \text{HV}_{0.3}$. This data demonstrates that the increased homogeneity and density of the $\alpha\text{-Al}_2\text{O}_3$ based coating contribute to its higher hardness compared to the coating based on $\gamma\text{-Al}_2\text{O}_3$ powder.

The study showed that the characteristics of the powder significantly affect the coating formation process. It is assumed that the temperature of the particles depends on their mass and thermal conductivity. Particles with greater mass require more heat to reach the desired temperature, making them more difficult to melt compared to smaller particles. The porosity of the particles also plays an important role in their thermal conductivity. Spherical particles with high porosity exhibit low thermal conductivity due to weak gas convection of stationary air in the pores.

According to Fig. 3, in coatings obtained by detonation spraying, different types of pores can be distinguished: large inter-lamellar, small inter-splat, and intra-splat pores. Large pores are formed due to incomplete filling of the gaps between previously deposited particles, which are likely not fully melted upon impact with the substrate/coating surface, and have a spherical shape. All coatings exhibit uneven pore distribution. Comparison of the coating structures (Fig. 3) shows that an increase in overall porosity is associated with the growth in both the size and number of pores. Coatings applied from spherical powder demonstrate the highest porosity (Fig. 4), indicating a greater number of large pores, whereas coatings from crushed powder have the lowest porosity (Fig. 4), with the fewest pores and a narrower size distribution. This phenomenon may be somewhat related to the morphology of the powder, as spherical powder contains more porosity than crushed fragmented powder. The change in hardness (Fig. 6) of the coatings is related to porosity, with mechanical properties at higher loads primarily depending on the coating structure. Overall, this analysis provides a clear understanding that the morphology of the initial powder affects the final microstructure and mechanical properties of the coatings.

4. CONCLUSIONS

The study showed that the detonation spraying method allows for the efficient application of coatings from two types of Al_2O_3 powders with different morphology (spherical and fragmentation) and phase composition ($\gamma\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$) but with similar particle sizes.

The study confirmed that when using optimized detonation spraying parameters, efficient coating application is ensured for both types of Al_2O_3 powders. At the same time, a difference in powder

utilization rates is observed: the coating obtained from the fragmentation form of α -Al₂O₃ powder demonstrates 65%, while spherical γ -Al₂O₃ particles allow for higher efficiency – 80%.

Coatings based on α -Al₂O₃ powder with a fragmentary particle shape have lower porosity (0.87%) and higher micro-hardness (1127 HV_{0.3}) compared to coatings made of spherical γ -Al₂O₃ powder (porosity 1.01%, micro-hardness 983 HV_{0.3}).

It was found that both phases are present in the studied Al₂O₃ based coatings: stable α -Al₂O₃ and metastable γ -Al₂O₃. This confirms that coating application, regardless of the morphology of the initial powder, does not lead to a complete phase transition from one form of aluminum oxide to another one, resulting in the formation of a mixed phase structure in the final product.

Microstructural analysis showed that the coating based on spherical powder has a less dense and uniform structure compared to the coating made of fragmented-particle powder. This is explained by the tendency of spherical particles to uneven distribution and void formation under impact during spraying, in contrast to fragmented-particles, which are more effectively packed, providing a denser and more uniform coating structure.

Thus, the morphology and phase composition of the powder are key factors influencing the technological parameters of coating application, as well as the microstructure and mechanical properties of the resulting coatings. The results of this work provide valuable information for various applications such as selecting the optimal powder type for specific coating conditions and requirements.

5. ACKNOWLEDGEMENTS

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