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Research of annealing influence on the hardness of detonation coatings from aluminum oxide

The article examines the effect of annealing on the structure and properties of alumina-based coatings obtained by detonation spraying. Coated samples were kept separately at temperatures of 500, 700, 800 and 1200 °C at a pressure of $3.6 \cdot 10^{-4}$ Pa for more than 1 hour. It was found that the microhardness of coatings made of alumina increases by 15-30 % after annealing depending on annealing temperature. The results of nanoindentation show that at 1200 °C the nanohardness of coatings after annealing increases by almost 100%. Aluminum oxide coating is characterized by high strength and density of the coating before and after annealing, and slight porosity. Results of X-ray analysis showed that the alumina powder consists of α -Al₂O₃ lattice, and after detonation injection coating cubes are converted into a semi- γ -cubic lattice. It was found that during the annealing of the coating at 1200 °C all cells of γ -phase completely transit to the α -phase. It was found that the increase in hardness after annealing of alumina coating at 500, 700, 800 and 1200 °C is associated with an increase in volume fraction of α -Al₂O₃ phase.

Keywords: detonation spraying, aluminum oxide, coating, microstructure, phase, microhardness, indentation, annealing.

Introduction

The surface of the piping elements of the boilers of thermal power plants or the blades of the steam turbines is often damaged by high temperatures and steam pressure. The damage caused by accidents results in significant damage to boiler parts. This has a direct impact on all other fields of society and creates a major economic crisis. Any (special) super alloys applied to the parts of the boiler corrode over time under the influence of high temperatures and pressures and lose their physical, mechanical and tribological properties. In order to prevent these shortcomings instead of inventing new materials in recent years scientists have focused on the development of new coating technologies that improve the surface properties of metals and alloys, and study the properties of the obtained coatings [1-4]. Coatings are widely used in automotive parts, boiler components, chemical process and medical equipment, aircraft engine elements, surface and offshore turbines, in the shipbuilding industry, etc. [5-7]. Thermal spraying, which is one of the technologies of surface treatment, is a cheap way to improve the surface properties of metal particles and to obtain a thick and high-quality coating. Among the thermal spraying methods are detonation spraying (DS) and high-velocity oxide spraying (HSOF), which allows to obtain a hard, dense and wear-resistant coating with excellent adhesive strength, low porosity and compressive residual stresses [8-10]. At the same time, the initial powder causes minimal oxidation of the material and provides high speed and relatively low

temperature [11, 12]. The sprayed powder particles adhere to the substrate at a speed of 800-1200 mps under the influence of detonation waves [13]. The quality of detonation coatings is directly related to the surface roughness of the pavement material (degree of processing), the chemical composition of the pavement material, the size of the granules, the ratio of gases that cause detonation explosions and impurities. TiO_2 , Cr_2O_3 , SiC , ZrO_2 , etc. were used to improve the properties or maintain the stability of $\alpha\text{-Al}_2\text{O}_3$ additives [14-16].

The low porosity of detonated coatings and the chemical composition of the initial powder in the coatings, as well as high adhesion strength of the coatings are relevant for the production of heat-resistant and heat-protective coatings on the blades of gas turbine engines. Aluminum oxide coatings are often used as layers of protection against high temperatures [17, 18]. The work on the study of alumina coatings obtained by detonation injection is not large and requires further study. It also allows to obtain the necessary properties for detonation heat-resistant coatings: coatings with high adhesive strength, low porosity, thickness up to 1 mm, as well as the ability to adjust the structure and properties of the coating by changing the technological parameters. Therefore there is great interest in the study of structural changes in the detonation coatings obtained from aluminum oxide during heat treatment. This work is designed to study the effect of thermal annealing on the structure and hardness of alumina coatings.

Materials and methods of research

Figure 1 shows a computerized complex of next-generation detonation spraying system CCDS2000 (detonation complex CCDS2000 (a) and its scheme (b)) [19-21]. The shaft is filled with gases by means of a high-precision gas distribution system and controlled by a computer. The process begins with 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 with the help of 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 m s^{-1} [22, 23].

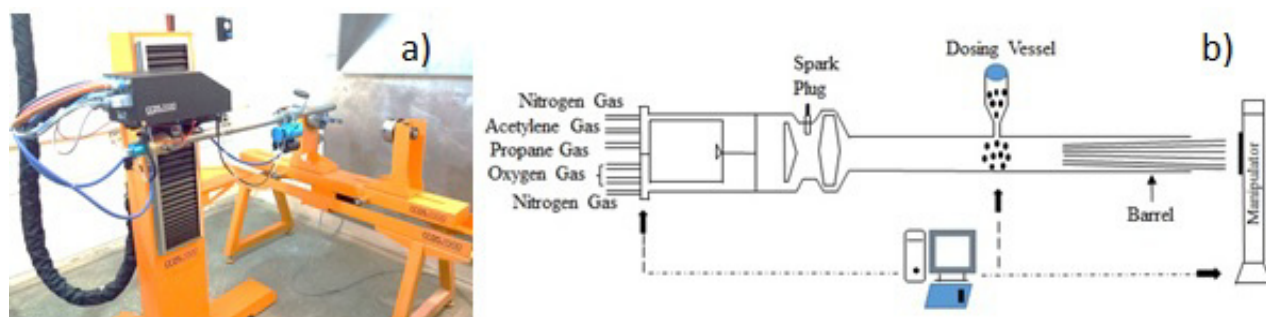


Figure 1. Computerized detonation complex CCDS200 (a) and its circuit diagram (b)

The method of detonation spraying was used to obtain Al_2O_3 coating. The size of the aluminum powder for spraying was $34 \pm 6 \mu\text{m}$. The injection shaft was placed on a detonation unit "CCDS-2000" with a diameter of 20 mm and a length of 800 mm (Figure 1). Stainless steel 12Ch18N10T was chosen as the lining material. Pipe elements of boilers of thermal power plants and elements of steam turbine engines are made of stainless chromium-nickel-based steels. These steels are often damaged by high temperatures and pressures. 12Ch18N10T steel is one of the universal steels operating in such aggressive environments. To protect the surface of 12Ch18N10T steel from corrosion, erosion and wear under the influence of high temperatures, the surface is coated with Al_2O_3 coating. Today it is the most used and widely used steel of all grades of stainless steel. That is why 12Ch18N10T steel was chosen as a substrate. The chemical composition of steel is in accordance with GOST 4986-79 [24].

The distance between the shaft and the sample was 250 mm. Samples of 70x50x3 mm with a roughness of the initial surface $R_a = 0.080 \mu\text{m}$ were selected as a substrate for spraying coatings. The sur-

face of the substrate was cleaned chemically for 5-7 minutes. To increase the average roughness of the dried substrates to 4.5 μm electrocorundum sand treatment with a grain size of approximately 300 μm was used. Thermal annealing of the coated samples was carried out in the laboratory in a vacuum electric oven SNVE-16/13 at a temperature of 500°C, 700°C, 800°C and 1200°C in a vacuum of 10^{-4} Pa for 1 hour. Metallographic analysis of the microstructure of coatings with the help of Altami MET 5C microscope and surface structure were studied with the help of electron microscopes scanning JSM-6390LV and PhenomProX. The microhardness of the samples was measured in accordance with GOST 9450-76 using a METOLAB instrument using a diamond indenter based on the micro-vickers method, holding at a load of 300 g for 10 s. The phase composition of the samples was studied on an X'PertPro diffractometer using X-ray diffraction analysis ($U = 40$ kv, $I = 30$ mA $\text{CuK}\alpha$). The obtained coatings with mechanical properties (Young's modulus, hardness) were studied by a NanoScan-4D Compact nanohardometer. The tests were carried out at a load of 200 mN. Loading time, unloading time and maximum load holding time were every 5 s. The dependence of the penetration depth during loading and unloading on the applied force was determined by the Oliver-Farr method. At least 10 measurements were made for each sample, and their results were averaged.

Research results and Discussion

The figure 2 presents the microstructure of the coatings before and after annealing. The thickness of the coatings was 300-500 μm . The coating has a porous structure. Figure 3 shows the microstructure of the surface of the coating; average size 10-15 μm .

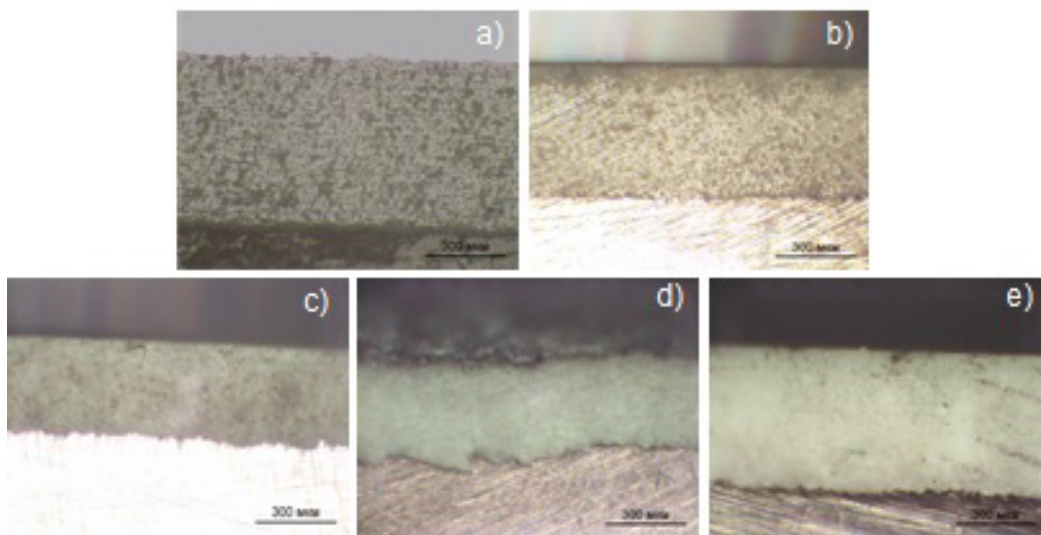


Figure 2. Microstructure of coatings from aluminum oxide before (a) and after annealing at 500°C (b), 700°C (c), 800°C (d) and 1200°C (e)

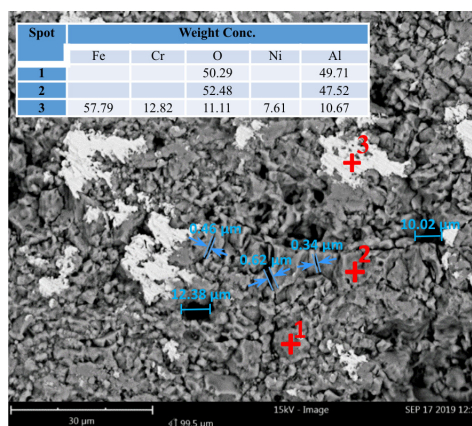


Figure 3. SEM-image of the surface

Figure 4 shows the dependence of the change in microhardness on the depth of the sample before and after annealing at different temperatures. The maximum increase in microhardness is observed in samples after annealing at 1200 °C. The maximum depth of the hardened layer for all coatings is 400 μm, i.e., it corresponds to the thickness of the coating.

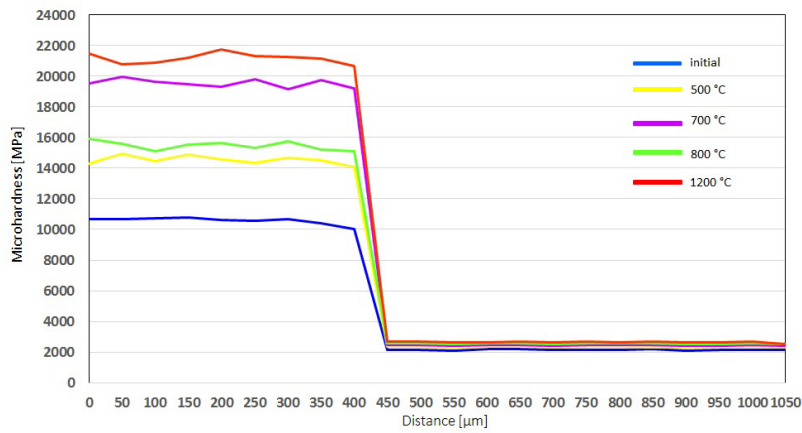


Figure 4. Microhardness of coatings from aluminum oxide

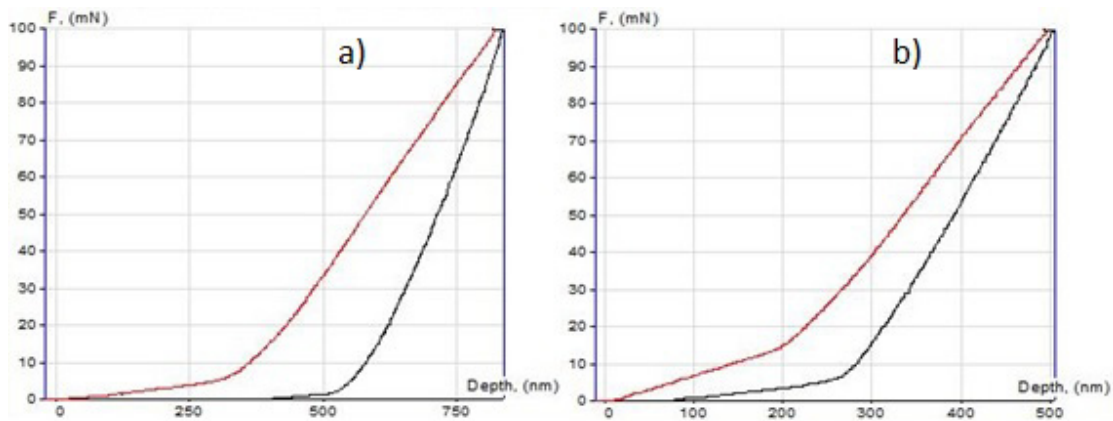


Figure 5. Nanoindentation curves of coatings from aluminum oxide before (a) and after annealing under 1200 °C

As a result of metallographic and microhardness analysis no thermal zone and diffusion zone are observed. During detonation injection the substrate is heated only to 200-300 °C, the surface of the substrate is not subject to structural and phase changes, and at the set annealing temperature there is no diffusion interaction between the alumina coating and the substrate. The nano-strength of coatings was also studied by the nano-indenting method (Figure 5). The relative curves of nanoindentification of the coating and its heat treatment are given. It can be seen that the penetration depth of the nanoindenter into the coating after annealing is 20% less than in the original coating.

On the nanoindentation study the modulus of elasticity with the nanohardness of coatings was determined (Table 1).

Table 1

Results of nanoindentation

Coatings	Nanohardness, GPa	Young's modulus, GPa	Pressure, Pa
Al ₂ O ₃ initial	10.7	186	3.6*10 ⁻⁴
Al ₂ O ₃ after annealing at 500 °C	14.3	224	
Al ₂ O ₃ after annealing at 700 °C	19.5	286	
Al ₂ O ₃ after annealing at 800 °C	15.9	238	
Al ₂ O ₃ after annealing at 1200 °C	20.8	297	

The results showed that the nanostability of the sample after annealing increases compared to the original. In addition, the maximum value of nanostability of 20.8 GPa is observed after annealing at 1200°C, which increased the modulus of elasticity of coatings by 111 GPa. This indicates a decrease in the elasticity and strength of the coatings. The increase in the nanohardness and elastic modulus during annealing at 1200°C can be explained by the complete transition of the γ -Al₂O₃ and α -Al₂O₃ phases formed after detonation injection to the α -Al₂O₃ phase (Figure 5).

Al₂O₃-based powder is capable of several modifications under the influence of high temperatures [25-28]. Also, during thermal injection the composition of the Al₂O₃ coating is α , γ , δ , θ and different phases, of which the formation of α and γ phases predominates. It is shown that these phases depend on the nature of the source material and the melting and freezing temperatures. R-3c consisted of a hexagonal α -lattice on the diffractogram line of aluminum powder in Figure 6 (a). After detonation injection it turns into a semi-cubic lattice (Figure 6 (b)), after annealing for 1 hour at 500°C (Figure 6 (c)) the intensity of phases α and γ decreases and the diffractogram lines widen by raising the temperature to 700°C for 1 hour. After annealing (Figure 6 (d)) the intensity of the α and γ -phases respectively increases slightly. After annealing for 1 h at 800°C (Figure 6 (e)), the lines of the α -phase did not change significantly, and the maximum expansion of the diffractogram line of the θ -(040) phase was observed at $2\theta = 46^\circ$. During annealing of the coating at 1200°C for 1 hour (Figure 6 (f)) all γ -phase grids are removed and completely transformed into α -phase, thinned in the diffractogram lines, the intensity is 4-5 times higher than in flame samples at 500, 700, 800°C. An increase in the volume fraction of the α -phase leads to an increase in hardness. It was possible to increase the physical and mechanical properties of the obtained coatings by heat treatment.

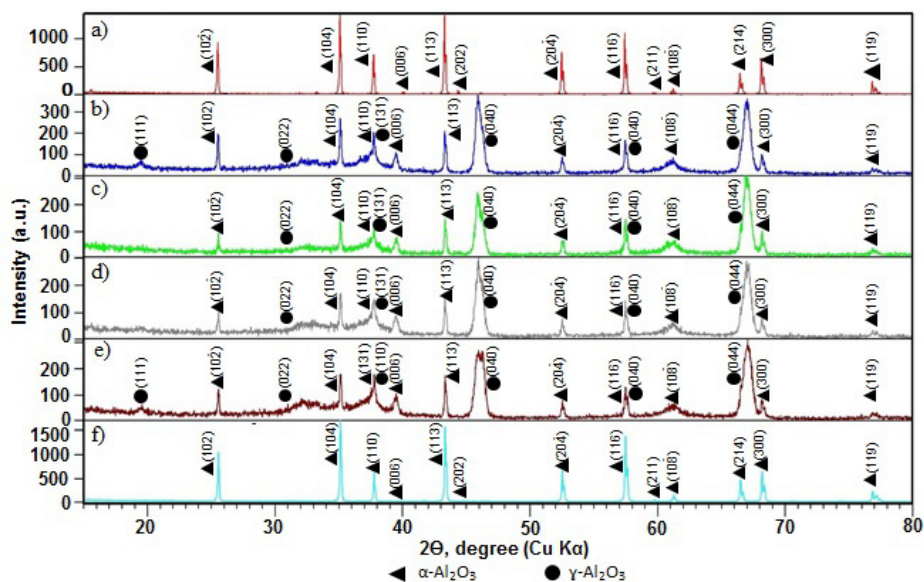


Figure 6. Diffractogram of Al₂O₃ powder in the initial and coating state after processing at different temperatures
a) Al₂O₃ powder b) Al₂O₃ coating c) 500°C d) 700°C e) 800°C f) 1200°C

Conclusions

1. Coatings of alumina with a thickness of 480-500 microns were obtained by detonation. The method of metallographic analysis revealed a decrease in the average size of cavities on the surface after annealing of the coating.

2. Raster electron microscopic analysis showed that the obtained coatings are characterized by high density and uniformity, as well as the presence of individual cavities. The cavities are found in two different states: round micro-cavities of several micrometers in size and micro-cavities in the form of thin layers, their length is 10-15 microns and thickness is 0.3-0.7 microns. After annealing there is a decrease in the number and size of micro-cavities in the form of thin layers.

3. X-ray structural analysis showed that the phase composition of the coatings before annealing consists of α -Al₂O₃ and γ -Al₂O₃ lattice. It was found that during annealing at 1200°C the α -Al₂O₃ phase is completely re-transferred.

4. It was found that the micro-hardness of alumina coatings after annealing increases by 15-30% depending on the annealing temperature. The results of nanoindentation showed that after annealing at 1200°C, the nanostability of coatings increases by almost 100% and reaches 20.8 GPa.

5. It was found that the increase in hardness during heat treatment of the coating is associated with an increase in the proportion of α -Al₂O₃.

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Күйдiрудiң алюминий оксидiнен жасалған детонациялық жабындардың қаттылығына әсерiн зерттеу

Мақалада детонациялық тозаңдау әдiсiмен алынған алюминий оксидi негiзiндегi жабындардың құрылымы мен қасиеттерiне күйдiрудiң әсерi зерттелген. Жабыны бар үлгiлер 500, 700, 800 және 1200°C температураларында жеке-жеке 1 сағаттан астам вакуумда $3.6 \cdot 10^{-4}$ Па ұсталды. Алюминий оксидiнен алынған жабындардың микроқаттылығы күйдiргеннен кейiн күйдiру температурасына байланысты 15-30 %-ға артатыны анықталды. Наноиндентрлеу нәтижелерi 1200°C кезiнде күйдiруден кейiнгi жабындардың наноқаттылығы екi есеге дейiн артатынын көрсеттi. Алюминий оксидi жабынының күйдiруге дейiнгi және одан кейiнгi жабын жоғары берiктiк пен тығыздыққа және аздап кеуектi болуымен сипатталған. Рентген талдау нәтижелерiнде, алюминий оксидi ұнтағының α -Al₂O₃ тордан тұратынын, детонациялық бүркуден кейiн жабынның текшелерi жартылай γ -кубтық торға ауысатыны анықталған. Жабынды 1200°C-та күйдiру кезiнде γ -фазаның бүкiл торлары α -фазаға толықтай ауысатыны дәлелденген. Алюминий оксидi жабынын 500, 700, 800 және 1200°C кезiнде күйдiруден кейiн қаттылықтың жоғарылауы α -Al₂O₃ фазаның көлемдiк үлесiне байланысты екенi анықталды.

Кiлт сөздер: детонациялық бүрку, алюминий оксидi, жабын, микроқұрылым, фаза, микроқаттылық, индентирлеу, күйдiру.

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Исследование влияния обжига на твердость детонационных покрытий из оксида алюминия

В статье исследовано влияние отжига на структуру и свойства покрытий на основе оксида алюминия, полученных методом детонационного напыления. Образцы с покрытием выдерживали $3,6 \cdot 10^{-4}$ Па при температуре 500, 700, 800 и 1200 °C в вакууме более 1 ч в отдельности. Установлено, что микротвердость покрытий из оксида алюминия после отжига увеличивается на 15–30 % в зависимости от температуры отжига. Результаты наноиндентирования показали, что при 1200 °C нанотвердость покрытий после отжига возрастает почти в два раза. Покрытие оксида алюминия до и после отжига характеризуется высокой прочностью, плотностью и незначительной пористостью. Результаты рентгеновского анализа показали, что порошок оксида алюминия состоит из α -Al₂O₃, а после детонационного распыления α -Al₂O₃ частично переходят в γ -Al₂O₃. Кроме того, доказано, что при отжиге покрытия при 1200 °C все решетки γ -фазы полностью переходят в α -фазу, а повышение твердости покрытия оксида алюминия после отжига при 500, 700, 800 и 1200 °C связано с увеличением объемной доли α -Al₂O₃ фазы.

Ключевые слова: детонационное напыление, оксид алюминия, покрытие, микроструктура, фаза, микротвердость, индентирование, отжиг.

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