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Research of annealing influence on the hardness of detonation coatings from zirconium dioxide

The article studied the effect of annealing on the structure and properties of zirconium dioxide coatings obtained by detonation spraying. Detonation spraying was realized on a computerized detonation spraying complex of the new generation CCDS2000. Thermal annealing of coated samples was performed at temperatures of 900 °C, 1000 °C, and 1100 °C. It was determined that the microhardness of zirconium dioxide coatings increases by 10–25 % depending on the annealing temperature after annealing. The results of nanoindentation showed that the nanohardness of the coatings after annealing at 1000 °C increases by 50 %. It was determined that after annealing at 1000 °C, the elastic modulus of the coatings increases, which indicates a decrease in plasticity and an increase in the strength of the coatings.. X-ray diffraction analysis showed that the phase composition of coatings before and after annealing consists of t-ZrO_2. After annealing occurs there is an increase in the degree of t-ZrO_2 tetragonality. Electron microscopic analysis showed that an increase in the number and size of micro-continuity in the form of thin layers after annealing. Determined that increase the hardness of zirconium dioxide after annealing at 900–1100 °C is associated with a higher degree of tetragonality t-ZrO_2 phase.

Keywords: zirconium dioxide, coating, detonation spraying, hardness, annealing, microstructure, phase, indentation.

Introduction

High-speed spraying methods can significantly expand the capabilities of traditional thermal spraying coatings used to protect parts from wear and corrosion [1–4]. Gas-thermal high-speed methods for producing coatings include methods of detonation [5], high velocity air-gas plasma (HVAGP) [6] and high velocity oil flame (HVOF) spraying [7]. Among them, the most promising is detonation spraying. Detonation spraying is one of the methods of thermal spraying of coatings, which is carried out using a special detonation gun filled with explosive gas mixture. A powdery spray material is used to form a coating. In the process of detonation, the particles of the powder are accelerated to high speeds (up to 1000 m/s), their melting and deposition on the sprayed surface [8].

The detonation method is promising for obtaining heat-resistant and heat-protective coatings on the blades of gas turbine engines due to the low porosity of the coatings and the saving of the chemical composition of the initial powder in the coatings, as well as the high adhesion strength of the coatings. Zirconium dioxide coatings are often used as upper thermal barrier layers of heat-protective coatings [9, 10]. There is very little work devoted to the study of zirconium dioxide obtained by detonation coatings. At the same time, detonation coatings allows one to obtain a set of properties necessary for heat-protective coatings: high adhesion of the coating, thickness up to 300 μm, significant porosity, as well as the ability to adjust the structure and properties of the coating by selecting processing parameters. Therefore, the study of structural transformations in detonation coatings of zirconium dioxide during heat treatments is of great interest. This work is devoted to studying the impact of thermal annealing on the structure and hardness of zirconium dioxide coatings.

Materials and methods of research

Detonation coatings were obtained on a computerized complex of new generation detonation spraying CCDS2000 (Computer Controlled Detonation Spraying), [11–14]. A general view and a schematic diagram of the detonation spraying process are presented in figure 1. 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. After that, a certain portion of the explosive mixture is supplied in

such a way that a layered gas medium is formed, consisting of an explosive charge and a carrier gas. Using a carrier gas stream, the powder is injected into the barrel (using a computer-controlled feeder) and forms a cloud. The substrate is placed at a certain distance from the exit from the trunk. After part of the powder is injected, the computer gives a signal to initiate detonation. This is realized 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. Particle velocities can reach 800 m s-1 [15–18].

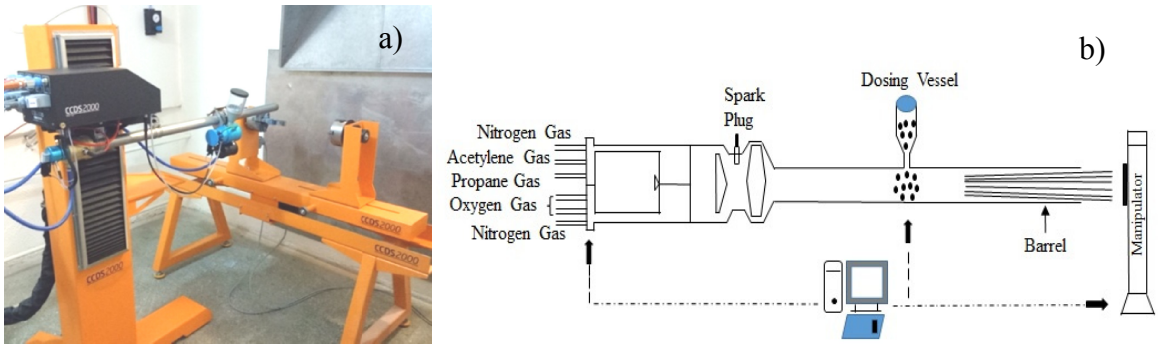

Figure 1. Computerized detonation complex CCDS200 (a) and its circuit diagram (b)

Sainless steel 12Cr18Ni10T was chosen as a substrate. The samples were sandblasted before coating. A powder of zirconium dioxide stabilized with yttrium oxide was used to obtain coatings. The particle size of the powder was up to 25–30 μm. Thermal annealing of the coated samples was carried out in a laboratory tube resistance furnace SUOL-0.4.4/12-M2-U4.2 in a vacuum of 10^{-2} Pa at temperatures of 900 °C, 1000 °C, and 1100 °C during 1 h. The temperature was measured and controlled by a VRT-2 precision thermoregulator using two thermocouples of the CCI 1378 type. The microstructure of the coatings was studied by metallographic analysis using a Neophot-21 microscope and scanning electron microscopy using JSM-6390LV and PhenomProX scanning electron microscopes. The microhardness of the samples was measured by the indentation method of a diamond indenter on a PMT-3 device in accordance with GOST 9450–76, at a load of 200 g and exposure under a load of 10 s. The phase composition of the samples was studied by X-ray diffraction analysis on an X'PertPro diffractometer using CuKα radiation. The measurement of hardness and elastic modulus was determined by the indentation method on a «NanoScan — 4D compact» nanohardness meter in accordance with GOST R 8.748–2011 and ISO 14577 indentation with a load of 0.1 N.

Research results and Discussion

The figure 2 presents the microstructure of the coatings before and after annealing. The thickness of the coatings was 360–370 μm. The coating has a porous structure. The average pore size is 5 μm.

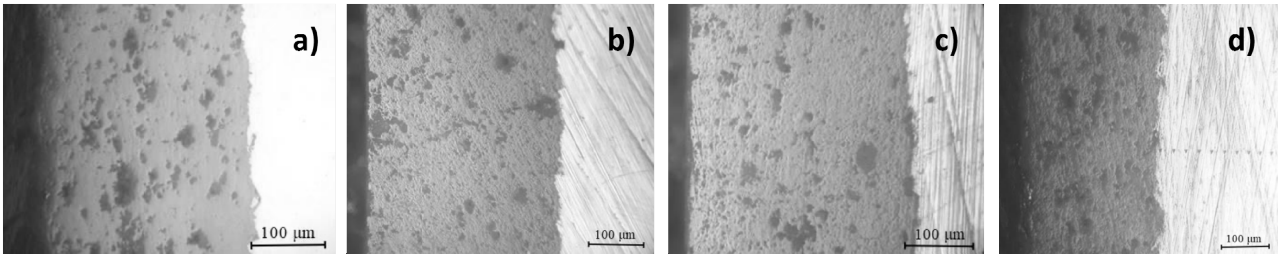

Figure 2. Microstructure of coatings from zirconium dioxide before (a) and after annealing at 900 °C (b), 1000 °C (c) and 1100 °C (d)

Figure 3 shows SEM-images of coatings and the results of X-ray microanalysis. The coating has a classic structure characteristic of gas thermal spraying methods. The coating is characterized by the presence of high density and uniformity as well as the presence of individual pores. Two groups of pores can be distinguished: rounded micro-discontinuities several micrometers in size and micro-discontinuities in the form of

thin interlayers, the size of which is several tens of micrometers in length and 0.3–1.0 μm in thickness. Thin layers are formed as a result of the spreading of molten particles of the sprayed metal over the surface. The results of X-ray microspectral analysis show that the formed coating is characterized by a more uniform distribution of all the chemical elements that make up the composition.

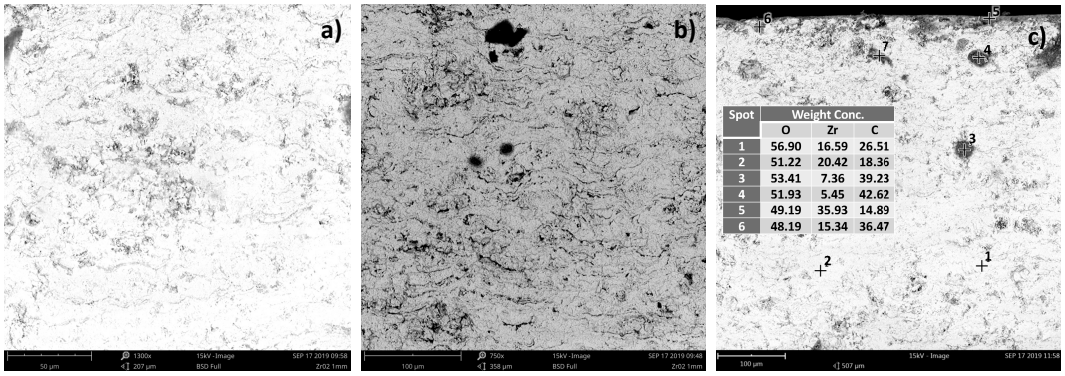

Figure 3. SEM-image of the surface (a), cross-section (b) of coatings
of zirconium dioxide and the results of micro X-ray spectral analysis (c)

Figure 4 shows the dependences of the microhardness variation along the depth of the experiment sample before and after annealing at different temperatures. The maximum increase in microhardness is observed in samples after annealing at 1000 °C. The maximum depth of the hardened layer for all coatings is 400 μm, i.e. corresponds to the thickness of the coating.

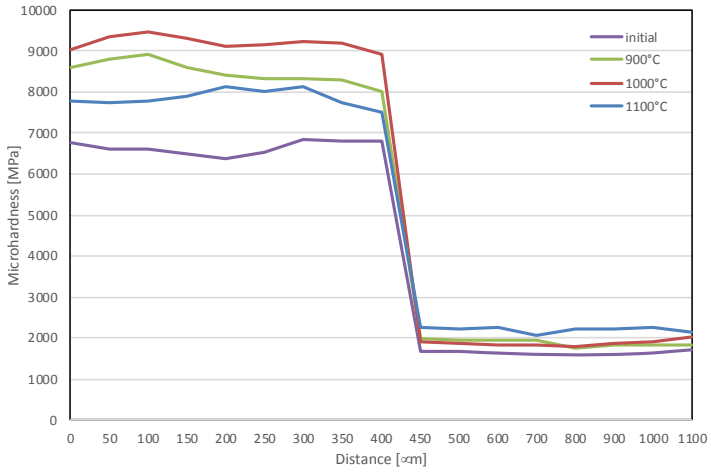

Figure 4. Microhardness of coatings from zirconium dioxide

The region of thermal influence and the diffusion zone are not observed according to metallographic analysis and microhardness. This is due to the fact that during detonation sputtering the substrate heats up to only 200–300 °C, that the surface of the substrate does not undergo structural and phase transformations, and also during annealing the diffusion processes do not occur between the zirconium dioxide coating and the iron-based substrate at the indicated temperatures.

We also studied the nanohardness of coatings by the nanoindentation method. Figure 5 presents the comparative nanoindentation curves for coatings before and after annealing. It is seen that the penetration depth of the nanoindenter into the coatings is 10 % less than the initial coating after annealing.

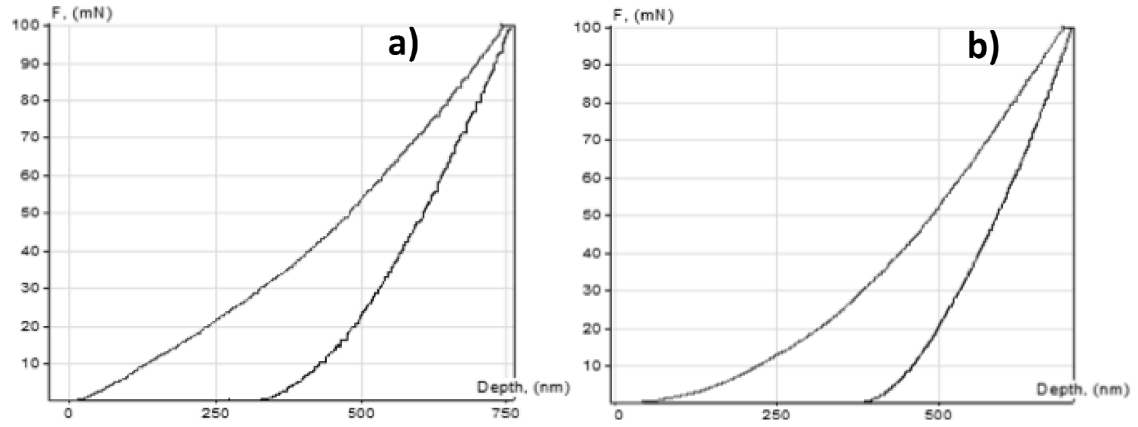

Figure 5. Nanoindentation curves of coatings from zirconium dioxide before (a) and after annealing under 900 °C (b)

The modules of elasticity and nanohardness of the coatings were determined on the results of nanoindentation (table 1). The results showed that the nanosolidity increases in comparison with the sample before and after annealing. In this case, the highest value of the nanohardness of 15.8 GPa is observed after annealing at 1000 °C. It can be seen that after annealing at 1000 °C, the elastic modulus of the coatings increases, which indicates a decrease in plasticity and an increase in the strength of the coatings.

Table 1

Results of nanoindentation

Coatings	Hanohardness, GPa	Young's modulus, GPa
ZrO_2 initial	9,9	176
ZrO_2 after annealing at 900°C	11,6	178
ZrO_2 after annealing at 1000°C	15,8	245
ZrO_2 after annealing at 1100°C	12,8	174

We can note a clear discrepancy (1.5 times) in the quantitative values of the results with good qualitative agreement by comparing the results of determining the hardness of the material at different loads on the indenter (microhardometry (figure 4) and nanosolidometry (table 1)). This can be explained by the fact that during nanocontact interaction, due to the small (tens of nanometers) dimensions of the indent, the degree of imperfection of the material under the indent is significantly reduced, which helps to bring the behavior of real material closer to ideal [19].

Figure 6 shows the diffraction patterns of the coatings before and after annealing. The results of x-ray structural analysis of coatings showed that the coating in the initial and after annealing consists of the t-ZrO_2 phase. The diffractogram of samples after annealing differs from the diffractogram before annealing in that instead of single lines (211) and (222), the t-ZrO_2 phase gives double lines. Also, after annealing the pairs of closely spaced each other lines (002) — (110) and (004) — (103), the t-ZrO_2 phases are moved wider apart. All this is related an increase in the tetragonality of the t-ZrO_2 phase. So as known [20] that the distance between paired lines depends on the c/a ratio. The larger it is, i.e., the greater the degree of tetragonality, the paired lines are further apart each other. In turn, the degree of tetragonality depends linearly on the oxygen content of zirconium dioxide. In our case, an increase in the degree of tetragonality after annealing due to an increase in the oxygen content is quite possible, since the annealing of the samples was carried out in a low vacuum.

Based on x-ray diffraction analysis, it can be claimed that the increase in the hardness of zirconium dioxide after annealing is associated with an increase in the tetrogonality of the t-ZrO_2 phase. Since the greater the degrees of tetragonality of the tetragonal phase, the higher the strength of the material [21].

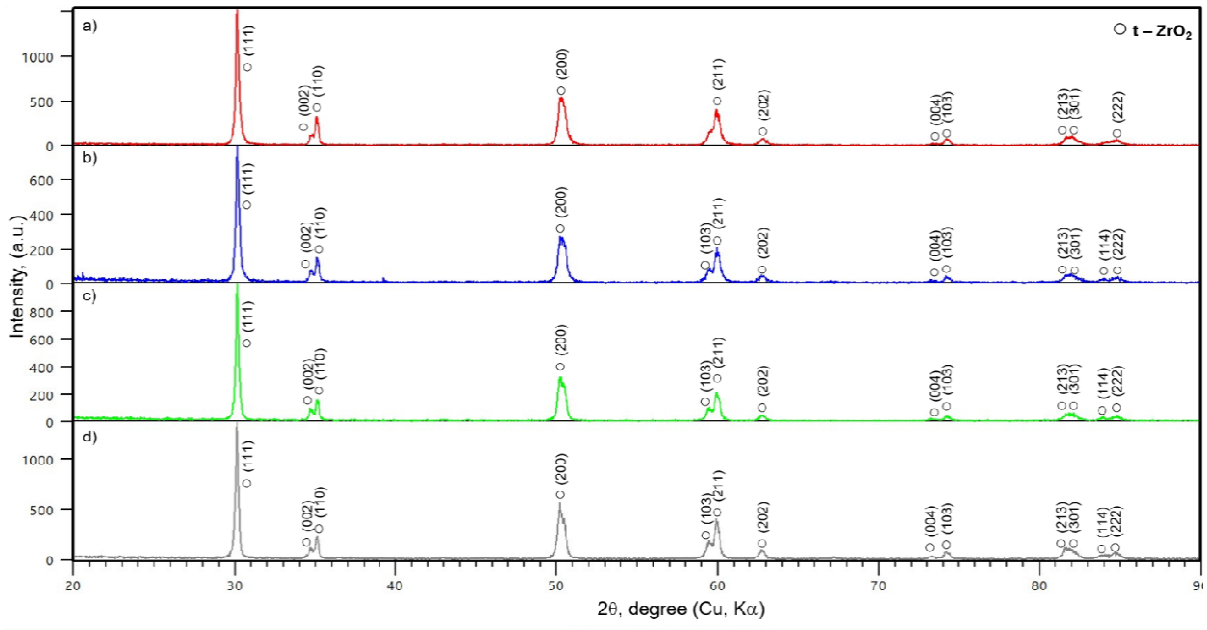

Figure 6. X-ray diffraction patterns of coatings from zirconium dioxide before (a) and after annealing at 900 °C (b), 1000 °C (c) and 1100 °C (d)

It can be seen on figure 7 that thermal extraction at 1000 °C based on structural influence is not provided. However, an increase in the number and size of micro continuities in the form of thin layers is observed. This helps to reduce internal stresses associated with operation. The formation of micro continuities in the form of thin interlayers is the reason for the strong discrepancy in the data on the microhardness and nanosolidness of the coating.

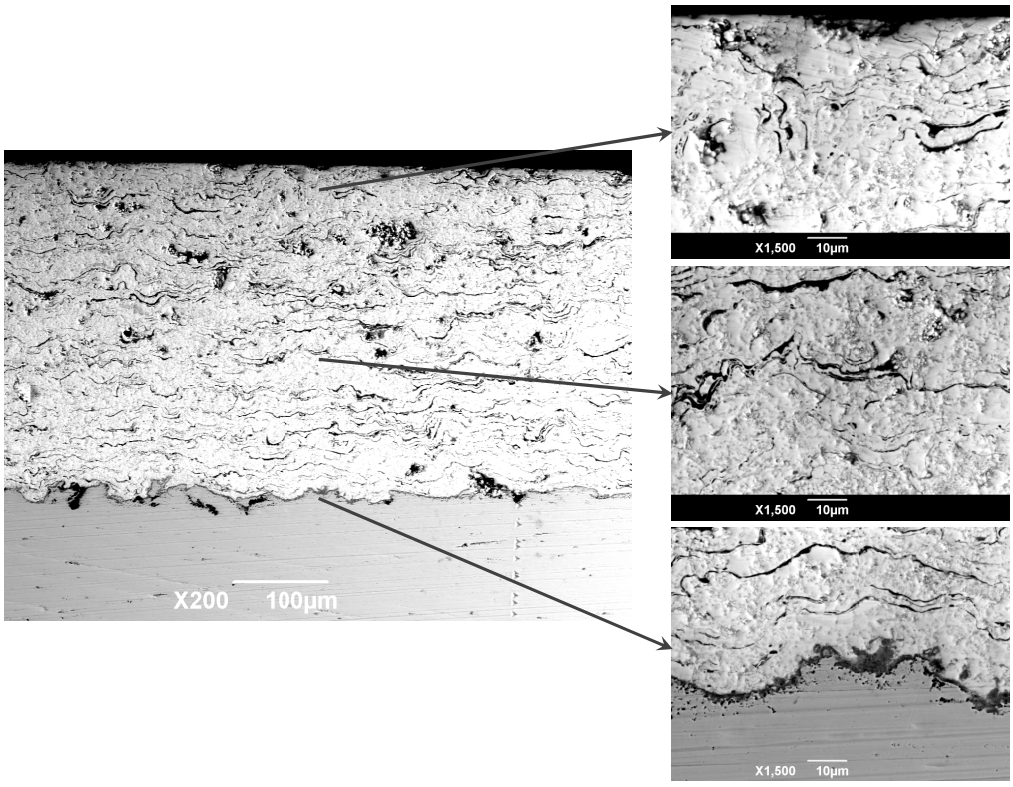

Figure 7. SEM-images of coatings from zirconium dioxide after annealing at 1000 °C

Conclusions

1. The coatings of zirconium dioxide with a thickness of 360–370 μm were obtained by the detonation method. It was determined that the coatings have pores and the average pore size is 5 μm by metallographic analysis method.

2. Electronic microscopic analysis showed that the resulting coatings are characterized by the presence of high density and uniformity, as well as the presence of individual pores. Two groups of pores have been identified: round micro-discontinuities several micrometers in size and micro-discontinuities in the form of thin interlayers, the size of which is several tens of micrometers in length and 0.3–1.0 μm in thickness. There is an increase in the number and size of micro-continuity in the form of thin layers after annealing.

3. X-ray diffraction analysis showed that the phase composition of coatings before and after annealing consists of t-ZrO_2. After annealing, there is an increase in the degree of t-ZrO_2 tetragonality.

4. It was determined that the microhardness of zirconium dioxide coatings increases by 10–25 % depending on the annealing temperature after annealing. The results of nanoindentation showed that the nanohardness of the coatings after annealing at 1000 °C increases on 50 % and reached to 15.8 GPa.

5. Determined that increase the hardness of zirconium dioxide after annealing at 900–1100 °C is associated with a higher degree of tetragonality t-ZrO_2 phase.

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Б.К. Рахадилов, Д.Н. Кәкімжанов, Н. Қантай, П. Ковалевский, Р.С. Қожанова

Цирконий диоксиді негізінде детонациялық жабындардың қаттылығына күйдірудің әсерін зерттеу

Мақалада детонациялық тозаңдату әдісімен алынған мырыш диоксиді негізінде жасалған жабындардың құрылымы мен қасиеттеріне күйдірудің әсері зерттелген. Детонациялық тозаңдандыру CCDS2000 жаңа буынды детонациялық тозаңдандыру компьютерленген кешенінде жүзеге асырылған. Қаптамасы бар үлгілерді термиялық күйдіру 900°С, 1000°С және 1100°С температураларында жүргізілген. Мырыш диоксидінен жасалған жабындардың микроқаттылығы күйдіргеннен кейін күйдіру температурасына байланысты 10–25 %-ға артатыны анықталды. Наноиндентірлеу нәтижелері, 1000°С кезінде күйдіруден кейінгі жабындардың наноқаттылығы 50 %-ға артатынын көрсеткен. 1000°С кезінде күйдіргеннен кейін жабындардың серпімділік модулі ұлғаяды, бұл жабындардың иілгіштігінің азаюы мен беріктігінің артуы анықталған. Мырыш диоксидінен күйдіруге дейінгі және одан кейінгі жабын жоғары тығыздықпен және біртектілігімен, кеуекті болуымен сипатталған. Рентгенқұрылымдық талдау нәтижелері, күйдіргенге дейін және одан кейін жабынның фазалық құрамы t-ZrO_2-ден тұратынын көрсеткен. Күйдіргеннен кейін тетрагональдік t-ZrO_2 деңгейінің өсуі байқалған. Электронды-микроскопиялық талдау нәтижелері күйдіргеннен кейін жұқа қабаттар түріндегі микроталшықтың мөлшері мен саны ұлғайғанын көрсетті. Мырыш диоксидінің 900–1100°С кезінде күйдіргеннен кейін қаттылығының жоғарылауы t-ZrO_2-фазасының тетрогональдік дәрежесінің жоғарылауымен байланысты екені анықталған.

Кілт сөздері: мырыш диоксиді, жабын, детонациялық тозаңдату, қаттылық, күйдіру, микроқұрылым, фаза, индентирлеу.

Б.К. Рахадилов, Д.Н. Какимжанов, Н. Кантай, П. Ковалевский, Р.С. Кожанова

Исследование влияния отжига на твердость детонационных покрытий из диоксида циркония

В статье изучено влияние отжига на структуру и свойства покрытий из диоксида циркония, полученных методом детонационного напыления. Детонационное напыление осуществляли на компьютеризированном комплексе детонационного напыления нового поколения CCDS 2000. Термический отжиг образцов с покрытием проводили при температурах 900, 1000 и 1100°С. Определено, что после отжига микротвердость покрытий из диоксида циркония увеличивается на 10–25 % в зависимости от температуры отжига. Результаты наноиндентирования показали, что нанотвердость покрытий после отжига при 1000°С повышается на 50 %. Определено, что после отжига при 1000°С модуль упругости покрытий увеличивается, что указывает на уменьшение пластичности и повышение прочности покрытий. Покрытие из диоксида циркония до и после отжига характеризуется наличием высокой плотности и однородности пор. Электронно-микроскопический анализ показал, что после отжига происходит увеличение количества и размеров микросплошности в виде тонких прослоек. Рентгеноструктурный анализ показал, что фазовый состав покрытий до и после отжига состоит из t-ZrO_2. После отжига наблюдается увеличение степени тетрагональности t-ZrO_2. Определено, что повышение

твердости диоксида циркония после отжига при 900–1100°С связано с увеличением степени тетрагональности t-ZrO_2-фазы.

Ключевые слова: диоксид циркония, покрытие, детонационное напыление, твердость, отжиг, микроструктура, фаза, индентирование.

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