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Investigation of mechanical and tribological properties of NiCrAlY/ZrO_2-Y_2O_3 coatings obtained by detonation spraying

In this study, multilayer gradient NiCrAlY/ZrO_2-Y_2O_3 coatings obtained by detonation spraying in 1D (spot sputtering) and 2D (full-surface scanning sputtering) modes were investigated. The structure of coatings was analyzed using scanning electron microscopy and electron dispersion analysis, mechanical properties (hardness, modulus of elasticity) were determined using various techniques. The tribological characteristics of the coatings including abrasion resistance and coefficient of friction were also studied. It was determined that the coatings consist of alternating layers of NiCrAlY and ZrO_2-Y_2O_3, creating a multilayer gradient structure. It was found that the NiCrAlY layers act as a bonding element, providing interlayer adhesion, and the ceramic ZrO_2-Y_2O_3 layer serves as a thermal barrier protecting the substrate from high temperature loads. It was found that the coatings in 2D mode have high microhardness compared to coatings in 1D mode. It was determined that the hardness of coatings smoothly increases from the substrate to the surface layers due to the gradient increase in the content of ceramic materials. It was found that coatings obtained in 2D mode have better wear resistance and lower coefficient of friction compared to coatings in 1D mode, indicating the greater efficiency of coatings in dry friction conditions and their ability to prevent wear of the substrate material.

Keywords: thermal protection coating, gradient coatings, detonation spraying, tribological properties, microhardness.

Introduction

Modern industry faces the need to improve the thermal stability of various machine parts and mechanisms. This is especially relevant for the aviation industry, power engineering, chemical industry and other areas where materials are exposed to high temperatures, aggressive media and intensive mechanical wear. One of the promising methods of improving the performance characteristics of materials is the use of thermal protective coatings (TPC) [1–5]. A thermal protective coating usually consists of a metallic bonding layer and a ceramic top layer of zirconium dioxide stabilized with yttrium oxide (YSZ). YSZ is often used as a top layer for high-temperature applications such as turbine blades in jet engines due to its low thermal conductivity, excellent chemical stability, and high fracture resistance. The metallic bonding coating typically performs two main functions: 1) reduces the thermal mismatch between the YSZ and the substrate, and 2) protects the substrate from oxidation and corrosion. MCrAlY (M = Ni or Ni, Co) is a widely used alloy for these purposes [6, 7].

The main problem with the use of heat protective coatings is the difference in thermal expansion coefficients between the part and coating materials. At significant temperature gradients, this can lead to cracking of the coating and loss of its protective properties [8–11]. The mismatch in thermal expansion coefficients between the bonding layer and the ceramic topcoat can also cause coating failure during thermal cycling [12–15]. The binder layer generally has a higher coefficient of thermal expansion than the ceramic topcoat, resulting in stress accumulation and coating damage. To address this problem, multilayer gradient coatings have been developed to equalize the coefficients of thermal expansion between the layers. Multilayer gradient coatings offer numerous advantages over single materials due to their improved properties. Such coatings reduce thermal stresses by evenly distributing the thermal mismatch between the metal substrate and the ceramic top layer over a large number of layers, which increases the durability of the TPC.

Various methods are used to apply thermal protection coatings, among which the most common are electron beam physical vapor deposition (EB-PVD) and plasma spraying. EB-PVD method allows to create coatings with columnar microstructure, providing high resistance to thermal cycling and improved adhesion [16–18]. Plasma spraying allows the formation of coatings with a lamellar microstructure, which provides good thermal insulation properties and resistance to thermomechanical stresses. Although these methods have been used with great success, they are costly and time-consuming, and coating complexly shaped parts can be difficult or even impossible to achieve [19].

The detonation spraying (DS) method has recently attracted increasing attention due to its high versatility and efficiency in working with various materials. Detonation spraying allows to vary technological parameters and alternate powder feeding, which makes it possible to control the temperature of the powder and the applied coating. This makes it possible to regulate the structural-phase state of the coating material and obtain coatings with specified properties. Advantages of the method include low porosity of the coating, high bond strength with the substrate, minimal thermal effects, which allows avoiding thermal stresses and deformations even in thin-walled complex parts. Due to the low porosity and preservation of the chemical composition of the powder, as well as the possibility of application using two dispensers, the detonation method is promising for obtaining heat protective coatings [9]. In this regard, multilayer gradient coatings by detonation spraying have been developed in this work, and their mechanical and tribological properties have been considered.

The purpose of this work is to investigate the hardness and wear resistance of NiCrAlY/ZrO_2 based gradient detonation coatings.

Materials and methods of research

In this work, 12Kh18N10T stainless steel with dimensions of 20mm was chosen as the substrate. The surfaces of the substrate were sanded with MIRKA brand sandpaper to achieve a smooth and even surface and sandblasted on all sides in a Nordberd NS3 percussion chamber to improve adhesion and relieve surface stresses.

Table 1

Chemical composition of steel grade 12X18H10T

C	Si	Mn	Ni	S	P	Cr	Mo	V	Cu
0.1–0.15	0.17–0.37	0.4–0.7	> 0.3	> 0.025	> 0.03	0.9–1.2	0.25–0.35	0.15–0.3	> 0.2

Metro 233B (YSZ) and PNX20K20Y13 (NiCrAlY) powders were used to obtain gradient coating based on NiCrAlY/ZrO_2-Y_2O_3. The morphology of YSZ and NiCrAlY powders are shown in Figure 1 (*a*, *b*), respectively. YSZ powder has a particle size range of 20–45 μm, and the powder particles are spherical in shape. PNX20K20Yu13 (NiCrAlY) powder particles have an irregular shape with a particle size of 40 μm.

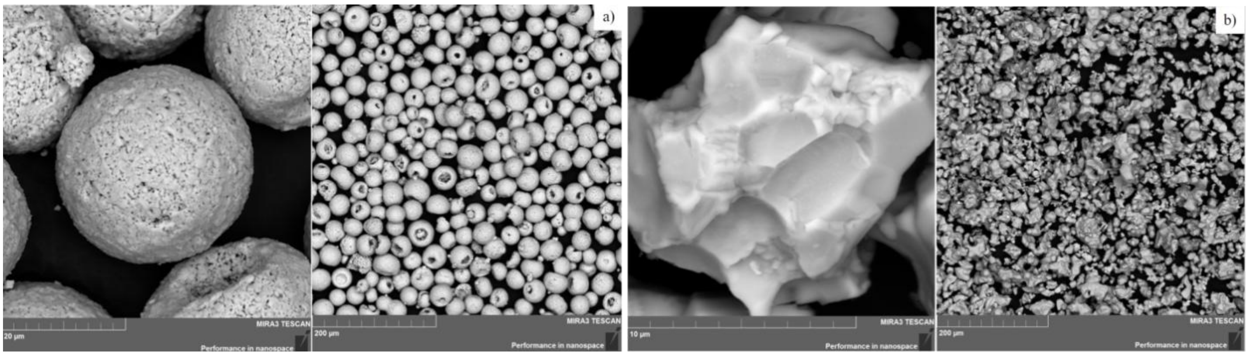


Figure 1. Morphology of powders YSZ (*a*), NiCrAlY (*b*)

The chemical composition of the powders is given in Table 2 and 2.1, respectively.

Table 2

Chemical composition of Metco 233B powder (YSZ)

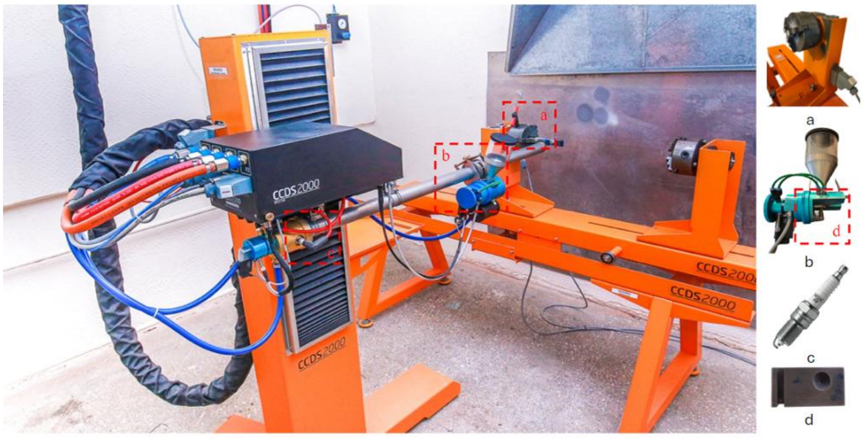
Y_2O_3 SiO_2 (max)	SiO_2 (max)	Al_2O_3 (max)	Fe_2O_3 (max)	TiO_2 (max)	Other oxides (max)	Monoclinic ZrO_2 (vol.% max)
7.0–9.0	0.5	0.2	0.2	0.2	0.8	<25

Table 2.1

Chemical composition of PNX20K20Yu13 powder (NiCrAlY)

Fe	Cr	Ni	Co	Mo	Al	Other (Y Si Nb C)
<0,3	20	basis	20	–	13	0.01–0.15

The CCDS2000 detonation spraying complex was used to produce gradient coatings. The coatings were applied to the samples using the CCDS2000 (computer-controlled detonation spraying) unit. Figure 1 shows the general view of the CCDS2000 detonation unit.



a — manipulator; *b* — powder dispenser; *c* — spark plug; *d* — dispensing plate

Figure 2. General view of the detonation unit CCDS2000

Gradient coating based on NiCrAlY/ZrO_2-Y_2O_3 were applied to 12Kh18N10T steel specimens using a method developed by us. This method includes abrasive blasting and coatings using PNX20K20Yu13 (NiCrAlY) and Metco 233B (YSZ) powders. The treated surface is infected with a jet of heated powder particles generated in the barrel of the detonation spraying unit. Surface blasting and coating are carried out sequentially in different detonation spraying model using the same NiCrAlY and YSZ powders. The surface blasting mode is selected so that the abrasive particles reach the sprayed surface in a solid state and atomize upon impact. The coating is applied by stepwise changing the detonation sputtering mode to obtain a gradient structure in which NiCrAlY smoothly transitions to YSZ from the substrate to the surface, and the sputtering process includes the following continuous steps:

– first stage — volume of barrel filling with gas mixture of acetylene and oxygen 35 % molar ratio of barrel filling O_2/C_2H_2 is 0.97;

– second stage — volume of barrel filling with gas mixture of acetylene and oxygen is 61 % molar ratio of barrel filling O_2/C_2H_2 is 2.52.

Gradient NiCrAlY/ZrO_2-Y_2O_3 coatings were obtained by detonation sputtering in two modes differing from each other in the number of shots and the form of application on the substrate: spot sputtering (1D), sputtering on the whole surface (2D). The number of shots in each of the modes is presented in Table 3 and Table 4.

Table 3

Number of shots for 1D mode

NiCrAlY	10	6	4	4	2	2	2	2	2	2	2	2	2	2	2	2
YSZ	2	2	2	2	2	2	2	2	2	2	2	2	4	4	6	40

Table 4

Number of shots for 2D mode

NiCrAlY	5	3	2	2	1	1	1	1	1	1	1	1	1	1	1	1
YSZ	1	1	1	1	1	1	1	1	1	1	1	1	2	2	3	20

The volume of barrel filling with burning mixture for the two modes was selected experimentally and is the same for both modes and is presented in Table 5.

Table 5

Volume of filling the detonation barrel with combustible mixture

Coating type	Molar ratio O_2/C_2H_2	Volume of barrel filling with gas mixture, %
PNX20K20Yu13 (NiCrAlY)	0.97	35
Metco 233B (YSZ)	2.52	61

The study of surface microstructure and analysis of morphology of cross sections of coatings were carried out using scanning electron microscopy (SEM) on MIRA3 TESCAN equipment and with the help of energy dispersive analyzer INCA ENERGY at E.A. Buketov Karaganda University.

The hardness tester “METOLAB 502” (GOST 6507-1-`2007) was used to check microhardness by Vickers method. The indenter used for measurement was a diamond pyramid with an angle between the two faces of 136º. The following mode was chosen to measure hardness by Vickers method: load 0.1 kg, load time 10 sec.

To measure the nanohardness and Young's modulus of the obtained coatings a hardness tester “FISHERSCOPE HM2000 S” was used, the principle of operation of which is based on the Martens method (DIN EN ISO 14577–1). For the hardness test a load of 2N and a dwell time at this load of 5 sec were chosen.

The obtained coatings with mechanical properties (Young's modulus, hardness) were investigated using a NanoScan-4D Compact nanohardness tester (FGBU “TISNSM”, Russia). Nanoindentation of coatings was carried out by Oliver and Farr method using Berkovich indenter at a load of 100 mN (ASTM E2546-07).

Studies of abrasive wear of samples were carried out on a special experimental stand in accordance with GOST 23,208-79, which corresponds to the American standard ASTM C6568 [20]. In the process of these tests, a technique based on the impact of a rotating roller on the flat surface of the specimen was used. Before starting the experiments, the specimens were pre-treated, including degreasing steps using acetone and subsequent drying. Then, a cylindrical rubber roller was pressed against the flat surface of the test specimen with a force of 44 N, and the roller began to rotate at a speed of 1 second per revolution. Abrasive particles (200–250 μm grit electrocorundum) were introduced into the test area at a rate of 41–42 g/min. The specimens were tested for 10 minutes, resulting in a total wear length of 28.8 mm.

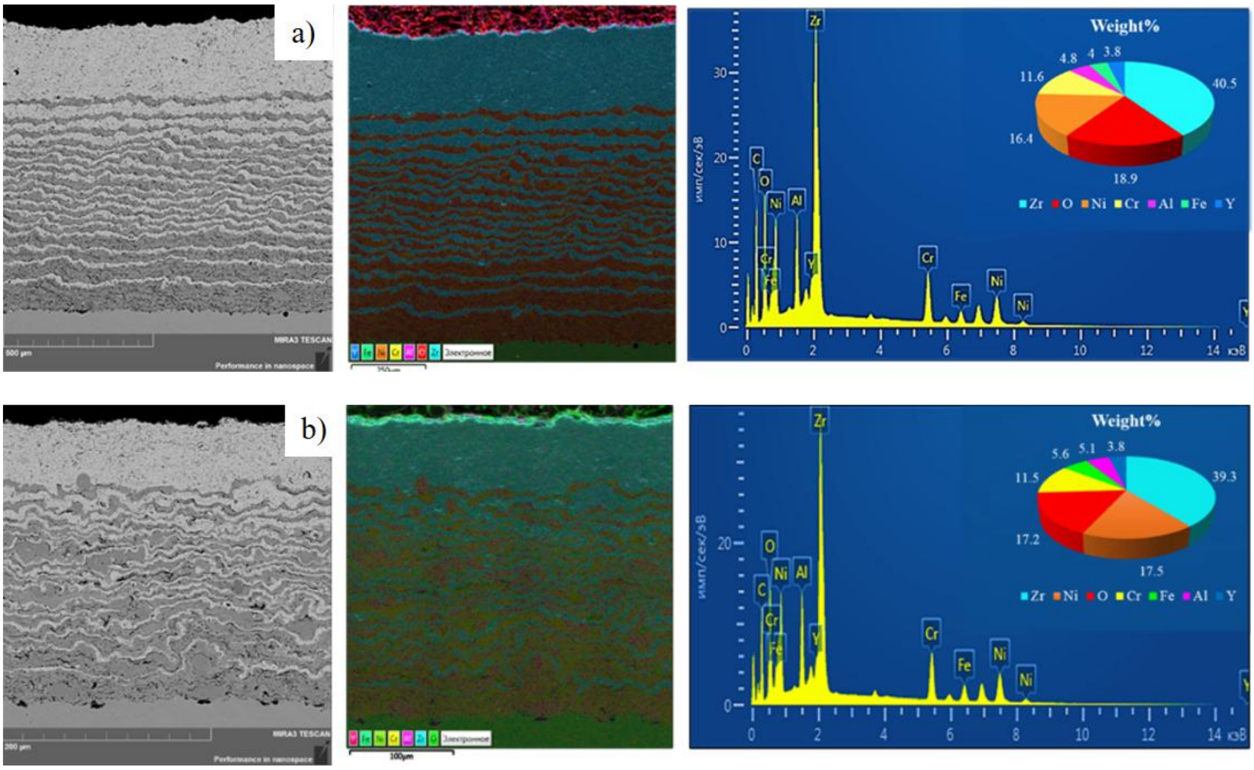
To assess the wear resistance of the tested samples, their wear was compared with the reference sample in accordance with GOST-23,208-79. The mass of samples was measured using analytical scales Gibertini CRYSTAL 100 CE with magnetic compensation. Before each weighing, the samples were cleaned from residual abrasive particles using compressed air. Assessment of material wear was carried out by measuring the change in mass of the samples during the tests in accordance with the requirements of GOST-23,208-79.

The reciprocating friction tests were performed using a TRB^3 tribometer from Anton Paar Srl, based in Peseux, Switzerland, using the standard ball-to-disk technique in accordance with ASTM G 133-95 and ASTM G99. A WC ball with a diameter of 6.0 mm was used as a counterbody. A load of 10 N and a full amplitude of motion of 5 mm with a frequency of motion of 7.50 Hz was selected. The length of the friction path was — 300 m. Then with the help of profilometer were measured the area of wear trace and by substituting the obtained values into the formula were calculated the values of wear volume, on the basis of the data obtained with the help of profilometer were created profilograms. Then, using specialized software, the coefficient of wear intensity for the studied coatings was calculated.

Results and Discussion

Scanning electron microscopy (SEM) results showed that the coating consists of alternating layers of NiCrAlY and ZrO_2-Y_2O_3. The ZrO_2-Y_2O_3 layer is located on the surface of the coating and acts as a thermal barrier layer, while the NiCrAlY layer serves as a bonding element between the substrate and the ceramic layer underneath the coating. The coating has a multilayer structure obtained by alternating layers of different materials. Several intermediate layers consisting of PNX20K20Yu13 and YSZ powders are present between the main layers. The electron dispersive spectral (EDS) analysis of the coatings in terms of the content of individual elements for the 1D mode is shown in Figure 3, and for the 2D mode in Figure 3.

The map of element distribution in the NiCrAlY/ZrO_2-Y_2O_3 gradient coating shows how the concentration of elements varies with the depth of the coating. This allows to evaluate the quality and homogeneity of the gradient transition between layers. The results of cross-sectional mapping of the studied samples showed the presence of the main elements of the coatings Zr, O, Ni, Cr, Al, Y and substrate Fe, with no extraneous impurities. The thickness of the layers varied depending on the number of detonation sputtering shots. The coating thickness was 273.72 ± 1.26 μm for sample 1D1 and 963.67 ± 13.59 μm for sample 2D2. Figure 3 also shows a gradual decrease in the NiCrAlY content from the substrate to the coating surface, while the YSZ content gradually increases. The study of the element distribution map confirmed that alternating layers of different materials helps to improve adhesion and reduce the probability of thermal stresses during coating operation. The thermal barrier properties of the coating due to the presence of the ZrO_2-Y_2O_3 layer provide effective protection of the substrate from high temperatures.



a — mode 1D; *b* — mode 2D

Figure 3. Element distribution map in NiCrAlY/ZrO_2-Y_2O_3 gradient coating

The results of hardness measurements by the Martens method on a Fisherscope HM 2000 hardness tester are presented in Table 6.

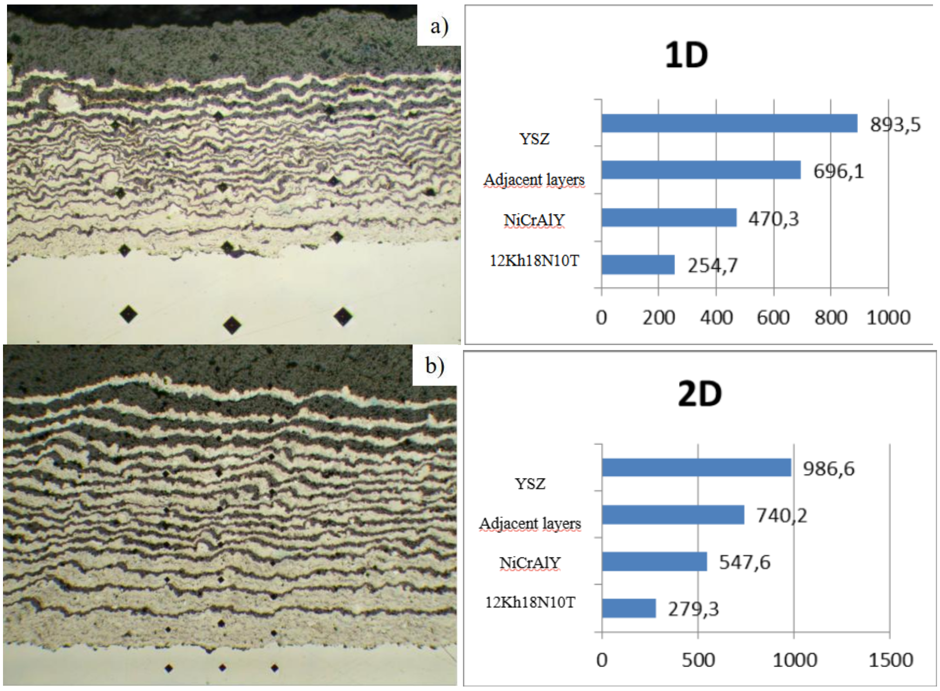
Table 6

Results of hardness measurements according to the Martens method

Mode 1D			Mode 2D		
№	HM [N/mm^2]	Eit/1–v^2 [GPa]	№	HM [N/mm^2]	Eit/1–v^2 [GPa]
1	3873.2	100.0	1	4658.3	111.1
2	4262.1	121.6	2	3398.4	87.9
3	3466.4	100.4	3	4153.9	109.5
4	4202.4	120.9	4	4733.0	122.0
5	3611.0	89.6	5	4853.3	129.8

The results show that the coated samples obtained using 2D mode have the highest hardness with an average hardness (HM) value of 4359.4 N/mm² and an average modulus of elasticity (Eit/1–v^2) of 112.1 GPa. In turn, the samples obtained using 1D mode have an average Martens hardness value of 3957.9 N/mm² and a modulus of elasticity of 108.6 GPa. The values for both samples are significantly higher than the hardness of the substrate material, which is 2002.03 N/mm² on the Martens scale. From these values, it can be concluded that these coatings significantly increase the hardness of the specimen. Investigation of the elastic modulus value showed that the original sample has a value of 186.86 GPa. The decrease in the elastic deformation ability of the sample is due to the presence of ZrO_2-Y_2O_3 ceramic layers in the coating.

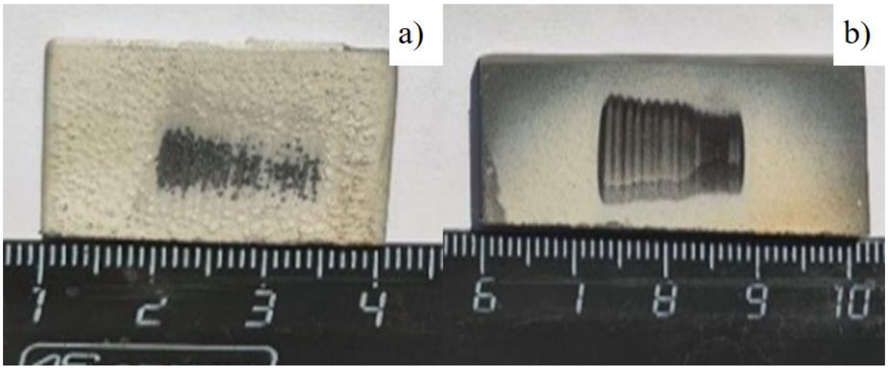
To determine the hardness of individual layers of the gradient coating, microhardness was investigated by the Vickers method [21]. The arithmetic mean of three measurements in each of the investigated layers was taken as the final values, since due to the design features of the device and the small thickness of the individual layers, the indenter trace was left on the bordering layers. The microindentation results show a clear pattern of increasing hardness values from the substrate to the YSZ surface layer. This trend is observed in both coatings. The hardness increases smoothly with each layer due to the gradient increase in ceramic content in the coating. The results of Vickers microhardness analysis for individual layers are presented in Figure 4. For convenience, cross-sectional images of two samples with indentation traces obtained with an Altamy Met 5C metallographic microscope were presented.



a — 1D mode; *b* — 2D mode

Figure 4. NiCrAlY/ZrO_2-Y_2O_3 gradient coatings in cross section

In addition to hardness determination, to study the protective properties of gradient coatings in two modes, the resistance to wear under dry friction against non-rigidly fixed particles was investigated according to GOST 23.208-79. The results of relative wear resistance were determined. According to the results of wear resistance studies the following values were obtained: the value of relative wear resistance for the 1D mode is 3 times higher than for the 2D mode and amounted to 0.97, while for the coating in the 2D mode this value amounted to 0.30. Figure 5 shows the footprints after WR measurement. The width of the trace for both modes was 1.5 cm. Visually, it can be seen that the abrasion test trace is deeper for mode 1D than for mode 2D, indicating the greater stability of the coating in mode 1D (Fig. 5).



a — 2D mode; *b* — 1D mode

Figure 5. Traces after relative wear resistance measurement

Figure 6 shows the time dependence of the friction coefficient of detonation multilayer gradient coatings. The average value of the friction coefficient of the sample in the first mode is 0.215 ± 0.048, and no coating failure was observed until the sliding distance reached 400 meters (or 4000 seconds). For the second specimen, the coefficient of friction was 0.584 ± 0.130. This value is higher than that of the first specimen, which may indicate its less effective thermal protection or sliding wear capability. The roughness and microhardness values can affect the tribological characteristics of the samples [22]. The increase in surface roughness of sample 1D compared to sample 2D leads to a decrease in the actual contact area of the interacting bodies, which also leads to a decrease in the coefficient of friction.

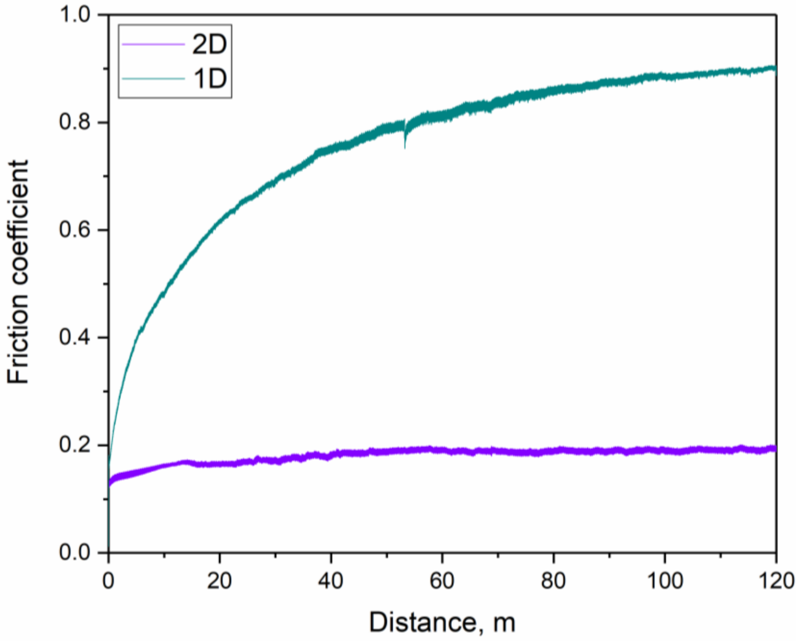
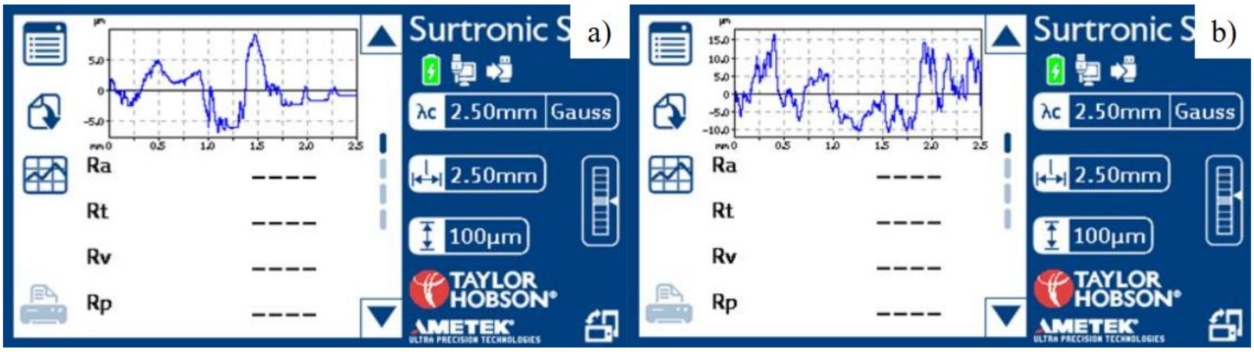


Figure 6. Graph of dependence of friction coefficient on friction path for both regimes

To determine the coefficient of wear intensity, profilograms were obtained on a Surtronic S128 abhvs TAYLOR HOBSON profilometer. According to the measurement results, the roughness for the sample with 1D mode was 4.62 μm, and for 2D mode — 5.23 μm. The profilograms are presented in Figure 7. The Kii value for the 1D sample was 5.226E-005 [mm³/N/m], for the 2D sample the same value was 6.805E-006 [mm³/N/m]. These parameters show the resistance of the obtained coatings to friction wear, as the results show the coating in 1D mode is more resistant to friction and wear.



a — mode 1D; *b* — mode 2D

Figure 7. Profilograms for modes

Conclusion

The following conclusions can be drawn from the studies of gradient multilayer NiCrAlY/ZrO_2-Y_2O_3 coatings obtained by detonation sputtering:

SEM and EDS analysis results confirmed the presence of a clear boundary between NiCrAlY and ZrO_2-Y_2O_3 layers, as well as intermediate layers consisting of PNX20K20Yu13 and YSZ powders. The elemental distribution map showed a homogeneous gradient transition between the layers, which is important for the thermal barrier properties of the coating. Hardness studies by the Martens method and microhardness by the Vickers method showed a significant increase in the hardness of the coated samples compared to the original substrate material. The samples obtained in 2D mode showed higher hardness and elastic modulus compared to those obtained in 1D mode. The results of wear resistance and coefficient of friction tests showed that the coatings obtained in 1D mode had higher wear resistance and lower coefficient of friction compared to those obtained in 2D mode. This indicates a better behavior of the 1D coating under dry friction and abrasion. Measurements of roughness and wear intensity coefficient showed that coatings obtained in 1D mode have lower roughness and higher wear resistance compared to coatings obtained in 2D mode.

In general, the developed NiCrAlY/ZrO_2-Y_2O_3-based gradient coatings exhibit high hardness, good adhesion, excellent thermal barrier properties and high wear resistance. These coatings can be effectively used to protect steel components operating under high temperature and abrasive wear conditions.

Acknowledgments

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Детонациялық бүрку әдісімен алынған $NiCrAlY/ZrO_2$-Y_2O_3 жабындарының механикалық-трибологиялық қасиеттерін зерттеу

Жұмыста 1D (бір орында нүктелік бүрку) және 2D (сканерлеу арқылы бүкіл бетке бүрку) режимдерінде детонациялық бүрку арқылы алынған $NiCrAlY/ZrO_2$-Y_2O_3 көп қабатты градиентті жылу қорғайтын жабындары зерттелген. Сканерлеуші электронды микроскопия (СЭМ) мен электронды дисперсиялық талдауды қолдана отырып, жабындардың микроқұрылымына талдау жүргізілді, әртүрлі әдістерді, соның ішінде Викерс әдісі мен Мартенс әдісін қолдана отырып, механикалық қасиеттері (қаттылық, серпімділік модулі) зерттелді. Сондай-ақ, жабындардың трибологиялық сипаттамалары, соның ішінде абразивті тозуға төзімділік және үйкеліс коэффициенті зерттеу жұмыстары жүргізілген. Жабындар NiCrAlY және ZrO_2-Y_2O_3 негізіндегі ауыспалы қабаттарынан тұратыны сондай-ақ, көп қабатты градиент құрылымын жасайтыны белгілі болды. NiCrAlY жабын қабаттары қабат аралық адгезияны қамтамасыз ететін байланыстырушы функцияны орындайтыны белгілі, ал ZrO_2-Y_2O_3 керамикалық қабаты төсенішті жоғары температура жүктемелерінен қорғайтын термиялық тосқауыл ретінде қызмет етеді. 2D режимінде алынған жабындар 1D режиміндегі жабындармен салыстырғанда жоғары микроқаттылықк мәнін көрсетті. Керамикалық материалдардың құрамының градиентті ұлғаюына байланысты жабындардың қаттылығы төсеніштен беткі қабаттарға дейін біркелкі өсетіні белгілі болды. 2D режимінде алынған жабындардың тозуға төзімділігі жоғары екені және 1D режиміндегі жабындармен салыстырғанда үйкеліс коэффициенті төмен екендігі анықталды, бұл зерттеу нәтижелері бойынша құрғақ үйкеліс жағдайында жабындардың үлкен тиімділігін және олардың төсеніш материалының тозуын болдырмау қабілетін көрсетеді.

Кілт сөздер: жылудан қорғайтын жабындар, градиент жабындары, детонациялық бүрку, трибологиялық қасиеттері, микроқаттылық.

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Исследование механико-трибологических свойств NiCrAlY/ ZrO_2–Y_2O_3 покрытий, полученных методом детонационного напыления

В статье изучены многослойные градиентные NiCrAlY/ZrO_2–Y_2O_3, полученные методом детонационного напыления в режимах 1D (точечное напыление в одном месте) и 2D (напыление на всю поверхность сканированием). Проведен анализ структуры покрытий с использованием сканирующей электронной микроскопии (СЭМ) и электронно-дисперсионного анализа, определены механические свойства (твердость, модуль упругости) с применением различных методик, включая метод Виккерса и метод Мартенса. Также изучены трибологические характеристики покрытий, включая абразивную износостойкость и коэффициент трения. Определено, что покрытия состоят из чередующихся слоев NiCrAlY и ZrO_2–Y_2O_3, создавая многослойную градиентную структуру. Установлено, что слои NiCrAlY выполняют функцию связующего элемента, обеспечивая межслоевую адгезию, а керамический слой ZrO_2–Y_2O_3 служит термобарьером, защищающим подложку от высоких температурных нагрузок. Установлено, что покрытия в режиме 2D обладают высокой микротвердостью по сравнению с покрытиями в режиме 1D. Определено, что твердость покрытий плавно увеличивается от подложки к поверхностным слоям благодаря градиентному увеличению содержания керамических материалов. Установлено, что покрытия, полученные в режиме 2D, обладают лучшей износостойкостью и более низким коэффициентом трения по сравнению с покрытиями в режиме 1D, что указывает на большую эффективность покрытий в условиях сухого трения и их способность предотвращать износ материала подложки.

Ключевые слова: теплозащитное покрытие, градиентные покрытия, детонационное напыление, трибологические свойства, микротвердость.

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