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INFLUENCE OF STRUCTURAL-PHASE CONDITION ON THE MECHANICAL-TRIBOLOGICAL PROPERTIES OF Ti_3SiC_2 COATINGS OBTAINED BY THE DETONATION METHOD

WPŁYW STRUKTURALNO-FAZOWEGO STANU NA WŁAŚCIWOŚCI MECHANICZNO-TRIBOLOGICZNE POWŁOK Ti_3SiC_2 UZYSKANYCH METODĄ DETONACJI

Key words: detonation spraying, titanium carbosilicide, heat treatment, microstructure, wear resistance.

Abstract

The article provides the results of the research of the structure and properties of powder coatings based on titanium carbosilicide Ti_3SiC_2 obtained by detonation spraying on the surface of tool steel U9/У9 (equivalent to N9). Micro-indentation methods and abrasive wear tests for mechanical and tribological properties of Ti_3SiC_2 based coatings were conducted. The microstructure of the coating has a layered structure. The border between the coating and the base has a characteristic crinkled appearance. It was determined that the phase composition of the coatings changes during detonation spraying is a result of the decomposition of Ti_3SiC_2 powder into titanium carbide and titanium carbosilicide (secondary phases). Selected consolidation conditions ensure the formation of a Ti_3SiC_2/TiC composite material. The influence of the second phase content (TiC, TiO_2) on the properties of coatings was studied. Studies of the microhardness of samples with coatings showed that, in the entire range of annealing temperatures, the microhardness of the Ti_3SiC_2/TiC composite material increases compared to the coating before annealing. It was found that the maximum microhardness of the Ti_3SiC_2/TiC composite material after annealing at a temperature of 800 °C is explained by an increase in the content of the Ti_3SiC_2 phase. It was established that, during detonation spraying of Ti_3SiC_2 powders, a coating with a higher microhardness and wear resistance is formed.

Słowa kluczowe: rozpylanie detonacyjne, karbosilikyd tytanu, obróbka cieplna, mikrostruktura, odporność na zużycie.

Streszczenie

W artykule przedstawiono wyniki badań struktury i właściwości powłok proszkowych na bazie karbosilikidu tytanu Ti_3SiC_2 uzyskanych przez rozpylanie detonacyjne na powierzchni stali narzędziowej U9/У9 (odpowiednik N9). Metody mikroindentacji i testy zużycia ściernego dla właściwości mechanicznych i tribologicznych powłok opartych na Ti_3SiC_2. Mikrostruktura powłoki ma strukturę warstwową. Granica między powłoką a podstawą ma charakterystyczny wygląd korby. Ustalono, że skład fazowy powłok zmienia się podczas rozpylania detonacyjnego w wyniku rozkładu proszku Ti3SiC2 na węglik tytanu i karbosilikyd tytanu (fazy wtórne). Wybrane warunki konsolidacji zapewniają tworzenie materiału kompozytowego Ti_3SiC_2/TiC. Zbadano wpływ zawartości drugiej fazy (TiC, TiO_2) na właściwości powłok. Badania mikrotwardości próbek z powłokami wykazały, że w całym zakresie temperatur wyżarzania mikrotwardość materiału kompozytowego Ti_3SiC_2/TiC wzrasta w porównaniu z powłoką przed wyżarzaniem. Stwierdzono, że maksymalna mikrotwardość materiału kompozytowego Ti_3SiC_2/TiC po wyżarzaniu w temperaturze 800°C tłumaczona jest wzrostem zawartości fazy Ti_3SiC_2. Ustalono, że podczas rozpylania detonacyjnego proszków Ti_3SiC_2 powstaje powłoka o wyższej mikrotwardości i odporności na zużycie.

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INTRODUCTION

An effective way to increase the reliability and durability of parts of various equipment and tools is to harden their surface by applying wear-resistant coatings. Among various methods of obtaining wear-resistant coatings, detonation spraying has several advantages **[L. 1]**. Its advantages are high adhesion strength (100–180 MPa) **[L. 2, 3]**, high density, and the possibility of obtaining coatings from most powders melting at temperatures up to 2800°C without decomposition. However, the successful implementation of this method is possible only with a rational choice of sprayed materials, which should be distinguished by high wear resistance and processability during spraying. Currently, widespread uses for the restoration of worn surfaces of a variety of products are found to be based on titanium carbosilicide. The Ti_3SiC_2 systems have good performance under abrasive wear, corrosion, and elevated temperatures, as well as relatively low cost **[L. 4, 5]**. In addition, interest in titanium carbosilicide as a unique composite material combining the properties of titanium carbide and titanium silicide has recently increased significantly. These substances are ternary compounds that correspond to the formula Mn+1AXn, where M is a transition metal, A is an element of IIIA or IVA groups, X is carbon or nitrogen, n = 1, 2 or 3 (they are also called MAX compounds). They have a hexagonal crystal lattice **(Fig. 1)**. A unique distinctive feature of these materials is the layered structure of their crystal lattice – the regular arrangement of the layers of atoms M and A elements (hence the name "nanolaminates"), which have a lower binding energy between them. There are many works devoted to the study of hardness and wear resistance of Ti_3SiC_2 coatings **[L. 6, 7**]; however, only a few of these coatings were obtained by the method of detonation sputtering. At the same time, various properties of coatings obtained by the method of detonation spraying and other methods are noted, making it of interest to study the tribological properties of the Ti_3SiC_2 detonation coating.

Due to the foregoing, the purpose of this work is to study the influence of the structural phase state on the tribological properties of Ti_3SiC_2 coatings obtained by detonation spraying on the surface of tool steel U9/У9.

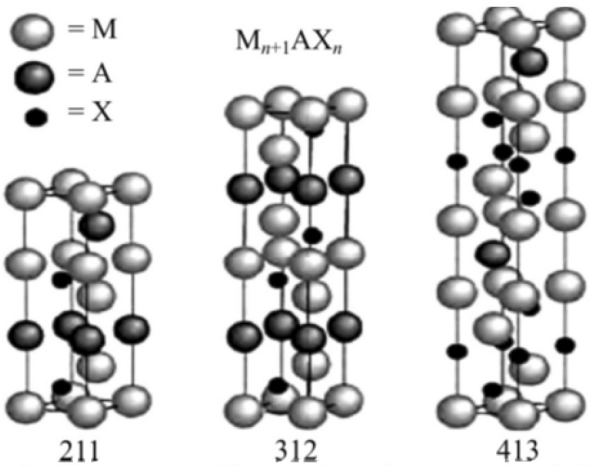
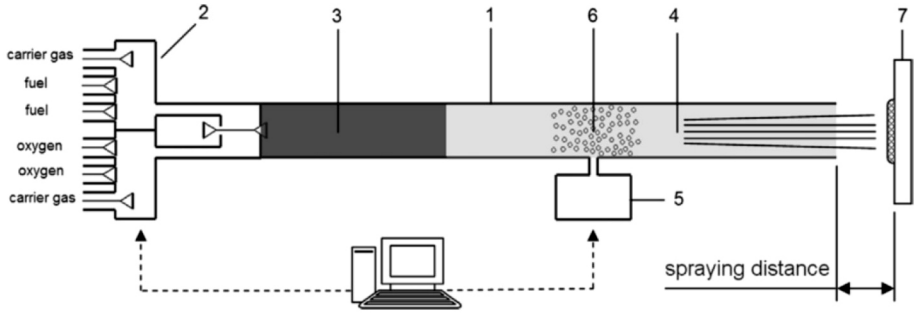


Fig. 1. The unit cell of the crystal lattice of titanium carbosilicide Ti_3SiC_2 [L. 6]

Rys. 1. Komórka elementarna sieci krystalicznej tytanokarbosilikonu Ti_3SiC_2 **[L. 6]**

MATERIAL AND METHODS

Detonation coatings were obtained on a computerized detonation spraying complex of a new generation of CCDS2000 (*Computer Controlled Detonation Spraying*) **[L. 8–10]**, in which detonation is realized inside the barrel in an explosive mixture formed as a result of flow-through supply of gas components through a specialized mixing device. A schematic of a CCDS2000 facility is presented in **(Fig. 2).** A distinctive feature of detonation spraying is a pulsed character of the process enabled by the procedure described below. A channel inside gun barrel (1) 850–1000 mm long and 20 mm in diameter is filled with gases by a computer-controlled precision gas distribution system (2); first, it is filled with a carrier gas, then with a certain portion of an explosive mixture, which results in the formation of a stratified gas medium consisting of explosive charge (3) and carrier gas (4). Then a certain amount of the feedstock powder is injected into the barrel through an orifice by a computer-controlled feeder (5) with the help of the carrier gas flow. The powder injected into the barrel forms a cloud (6), which for the taken barrel diameters and injection conditions can be considered uniform in density across the barrel cross-section. The cloud extends over a distance of up to 2–3 barrel diameters from the injection point having a concentration gradient along the barrel axis. The spraying distance is measured as a distance from the exit of the barrel (1) to the surface of substrate (7). Once the powder is injected, the computer gives a signal to initiate detonation, which is done by an electric spark. An explosive combustion of the charge occurs within a time of the order of 1 ms such that a detonation wave forms in the explosive mixture transforming into a shock wave in the carrier gas. The detonation products heated up to 3500–4500 K and the carrier gas heated by the shock wave up to 1000–1500 K move at a supersonic speed and exchange heat and interact with the powder during 2–5 ms. In this process, the powder particles can be heated up to the material's melting temperature and accelerated up to velocities as high as 500 m/s. Here, depending on the initial composition of the explosive mixture and the nature of the carrier gas, certain chemical reactions are possible causing changes in the phase composition of the sprayed powder. A CCDS facility is thus a dynamic reactor, in which, using controllable flows of chemically active gases, it is possible to induce chemical reactions in the sprayed powder materials and synthesize coatings containing newly formed phases – reaction products. **[L. 11, 12].**



1 – gun barrel, 2 – computer-controlled precision gas distribution system, 3 – explosive charge, 4 – carrier gas, 5 – computer-controlled powder feeder, 6 – cloud of injected powder, 7 – substrate.

Fig. 2. A schematic of a CCDS2000 facility [L. 11]
Rys. 2. Schemat instalacji CCDS2000 **[L. 11]**

As an object of research, instrumental carbon steel grade U9/У9 was selected,and the surface of which was previously subjected to sandblasting. The chemical composition of the powder is Ti -74 wt.%; SiC - 20 wt.%; C-6.0 wt.%. Thermal annealing of coated samples was carried out in a laboratory tubular electric resistance furnace of the SUOL-0.4.4/12-M2-U4.2 type in a vacuum of 10–2 Pa at temperatures of 700°C, 800°C, and 900°C for 1 h, with subsequent cooling. The temperature was measured and regulated by a precision temperature regulator VRT-2, using two thermocouples of the type of CCI 1378. The study of the surface morphology was performed by scanning electron microscopy using secondary (SE) and back-scattered electrons (BSE) on a Vega3 Tescan scanning electron microscope. The microhardness of the samples was measured by the method of the indentation of the diamond indenter on the device PMT-3 in accordance with GOST 9450-76, with a load of 200 g and an exposure under 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. Test samples for abrasive wear was performed on an experimental setup for testing for abrasive wear according to the scheme "rotating roller – flat surface" **[L. 13]** in accordance with GOST 23.208-79, that corresponds to the American standard ASTM S 6568. The durability of the treated sample was evaluated by comparing its wear with the wear of the reference sample (steel 45). The wear resistance of the test material was estimated by the weight loss of the samples during the test. Wear was measured by the gravimetric method on an ADB-200 analytical balance with an accuracy of 10^{-4} g. Tribological sliding friction tests were carried out on a tribometer THT-S-BE-0000 using the standard "ball-disk" technique (international standards ASTM G 133-95 and ASTM G 99) **[L. 14]**. As a counterbody, a VK ball with a diameter of 3 mm was used. The parameters of the studies were the same for the studied samples, i.e. the path length was 31 m, the load 10 N, speed 5 cm/s, at room temperature. The wear tracks were studied by using a contactless 3D profilometer of MICROMEASURE 3D station.

RESULTS OF RESEARCH

Research of the structure and properties of powder coatings based on Ti_3SiC_2

It is well known that the structure of coatings is determined by the mechanism of their formation. The main factors affecting the pattern of the formation of detonation coatings are heating, melting (full or partial), deformation, and spreading of particles over the surface. In contrast to the compact material, in the metal-coating composition, there are boundaries between deformed particles and layers **(Fig. 3a)**, as well as the boundary between the base metal and the coating **(Fig. 3a).** The border between the coating and the base has a characteristic zigzag appearance. Coating thickness is 1140 μm.

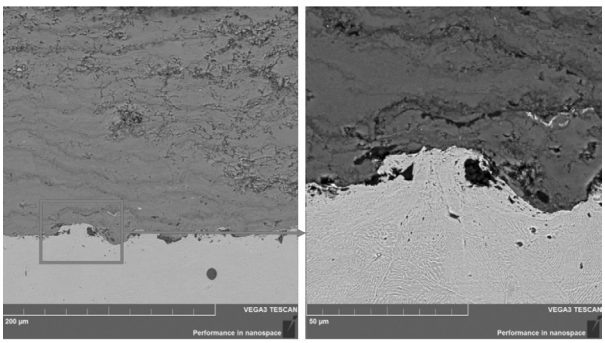


Fig. 3. Microstructure of the surface and cross-section of a U9/У9 steel sample coated with Ti_3SiC_2
Rys. 3. Mikrostruktura powierzchni i przekroju próbki stali U9 / У9 pokrytej Ti_3SiC_2

From the analysis of images, it follows that the coatings have a layered structure. Moreover, oxide films and pores between particles must inevitably be present in the structure itself. Using the EDS analysis, the elemental composition of the coatings and the base material was determined **(Fig. 4)**. It can be seen from the obtained data that the coating consists of elements Ti -62.82%, Si - 8.75%, and C-11.62%, and the structure of the coatings

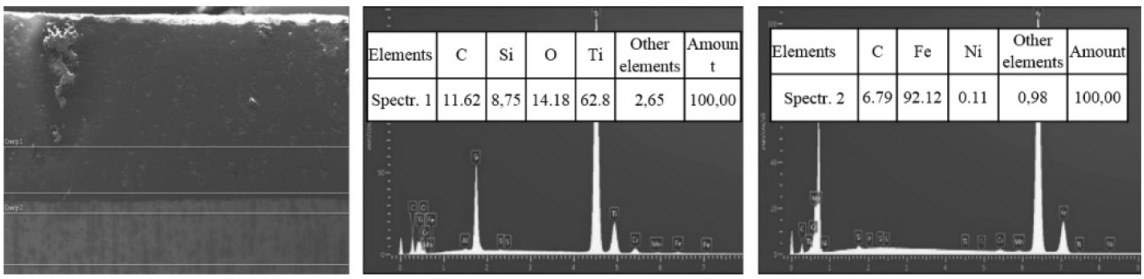


Fig. 4. Microstructure and elemental composition of the base material and coatings
Rys. 4. Mikrostruktura i skład pierwiastkowy materiału podstawowego i powłok

generally corresponds to the chemical composition of the powder Ti_3SiC_2, as well as detected oxygen O-14, 18%. As expected, all coatings have oxide films and pores. After detonation spraying, the chemical composition of the coating is preserved. The content of the following elements Fe -92.12% is visible from the second spectrum C-6.79%; which corresponds to the elemental composition of the base material.

Obviously, the study of the structure should be carried out in conjunction with the study of mechanical and tribological properties. This is necessary to control the quality of the sprayed layer to identify structural heterogeneity in the depth of the coating. **Figure 5** shows the changes in the microhardness of coatings over the depth of the layer.

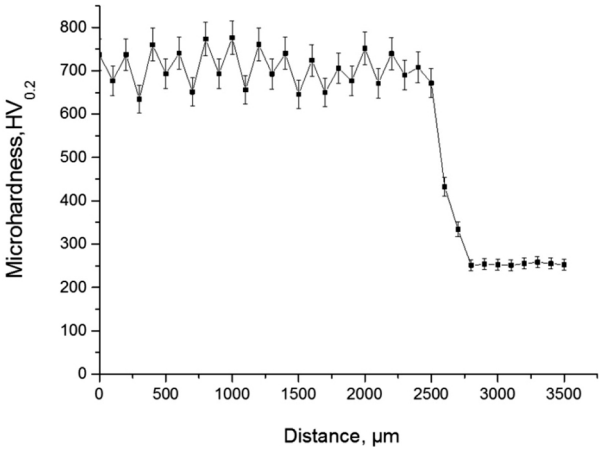


Fig. 5. Microhardness variation in the depth of the Ti_3SiC_2 coating layer
Rys. 5. Zmiany mikrotwardości w głębokości warstwy powłoki Ti_3SiC_2

Experimental data points have fairly stable limits of microhardness variation over the cross section of coatings. In this case, the upper limit of the microhardness value corresponds to the coating. From the given dependences, it is clear that the distribution is inherent in unevenness. This is due to the heterogeneity of coatings in composition and the presence of pores. A sharp drop in microhardness occurs at the interface of the coating/substrate - during the transition from the coating material to the steel base, where the structure and chemical composition of the material also changes dramatically. The Ti_3SiC_2 coating is characterized by increased microhardness (700 $HV_{0.2}$) compared to the substrate (250 $HV_{0.2}$). During detonation spraying of Ti_3SiC_2 powders, a coating with a higher microhardness is formed, compared to the base material (U9/У9).

We researched the phase composition of Ti_3SiC_2 coatings on the surface of U9/У9 and the initial powder. The results of X-ray phase analysis of coatings showed that the powder consists of Ti_3SiC_2 and TiC **(Fig. 6a)**. On the diffractogram of coatings **(Fig. 6b)**, a decrease in the intensity of the Ti_3SiC_2 diffraction lines and an increase in the intensity of TiC are observed, indicating a partial decomposition of titanium carbosilicide and is consistent with the data of **[L. 15]**. A decrease in the intensity of diffraction lines in titanium carbosilicide after detonation spraying, due to silicon deintercalation from the carbosilicide lattice layers **[L. 16]**, since silicon planes have weak bonds with TiC planes. This can be explained by the fact that as a result of detonation spraying, i.e. titanium carbosilicide lost some silicon due to its high volatility. Thus, the selected consolidation conditions ensure the formation of a Ti_3SiC_2/TiC composite material (*in-situ* formation of a composite). The advantage of such an

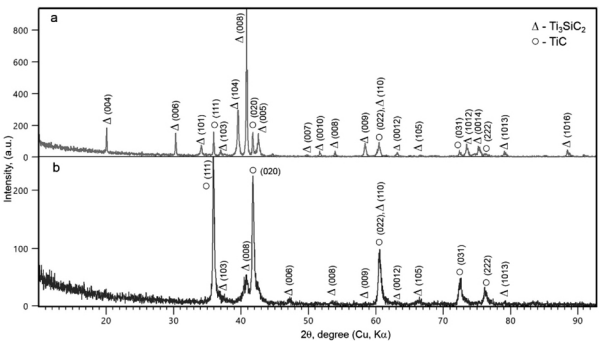


Fig. 6. Diffraction pattern of the samples studied: a) Ti_3SiC_2 powder; Ti_3SiC_2 coating on the surface of U9/У9 steel
Rys. 6. Wzór dyfrakcji badanych próbek: a) proszek Ti_3SiC_2; powłoka Ti_3SiC_2 na powierzchni stali U9/У9

in-situ composite is that the formation of titanium carbide TiC is an obligatory thermodynamically determined stage of the formation of the ternary compound Ti_3SiC_2 in the process of detonation spraying **[L. 6, 7, 15–18]**. As a result of detonation deposition of coatings in a detonation wave flow, the starting material of the powder undergoes a series of phase transformations as a result of the decomposition of Ti_3SiC_2 powder into TiC, due to the high-speed impact interaction heated to high temperatures.

For the coatings of this functional purpose, wear resistance is the most important operational property, the level of which affects both the performance of structures as a whole and the preservation of the geometrical dimensions of individual parts. The results of testing samples for abrasive wear were characterized by the loss of mass and coefficient of wear of the samples before and after the test. Reference samples were made of steel 45 according to GOST 1050-88. The wear of the test and reference samples was determined by weighing before and after testing with an error of no more than 0.1 mg. **Table 1** shows that the weight loss of the sample with the coating is less than that of the original sample, which indicates an increased resistance to abrasive wear. This is due to the presence in the coating composition of Ti_3SiC_2 of a larger share of hardening carbide phase TiC. Detonation coatings with bases of titanium carbosilicide are characterized with high wear resistance.

Table 1. The results of the test on the loss of mass and wear rate

Tabela 1. Wyniki testu utraty masy i szybkości zużycia

№	Name of samples	Microhardness, $HV_{0.2}$	Weight loss, mg	Wear coefficient, K
1	45	-	0.0302	1
2	U9/У9	250 ±32	0.0265	1,14 ±0.14
3	Coating Ti_3SiC_2	700 ±91	0.0222	1.37 ±0.17

Research of the mechanic-tribological characteristics of Ti_3SiC_2/TiC coatings after annealing

On the bases of the analysis of literature sources **[L. 4, 15–17]** and preliminary studies, it was suggested that, if detonation sputtering of the Ti-C-Si system was carried out, a multi-layer coating containing phases such as titanium carbides, silicides, and carbosilicides is possible during subsequent heat treatment with regulation of its phase composition. Thermal annealing of the coated samples was carried out in a laboratory tubular electric furnace at temperatures of 700°C, 800°C, and 900°C for 1 h, followed by cooling.

Figure 7 shows a plot of microhardness versus annealing temperature. It can be seen that the microhardness of the initial coating is 700 $HV_{0.2}$. After annealing, there is an increase in microhardness: at T = 700°C, the microhardness is 1150 $HV_{0.2}$, at T = 800°C, the microhardness is 1400 $HV_{0.1}$, and at T = 900°C, the microhardness is 850 $HV_{0.2}$. With the aim of identifying the reasons for the change of the microhardness, we have conducted X-ray phase analysis of the coatings before and after annealing.

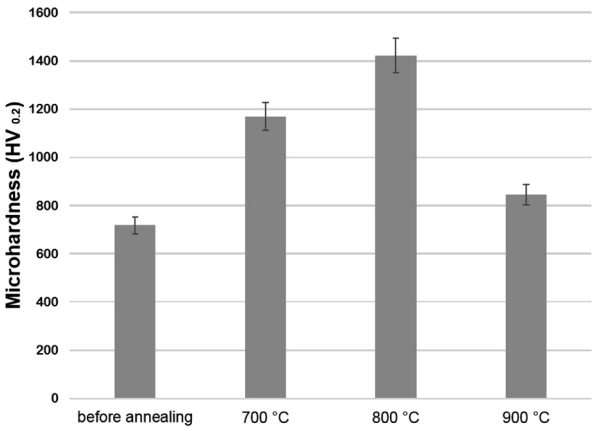


Fig. 7. The effect of annealing temperature on microhardness of coating based on Ti_3SiC_2/TiC

Rys. 7. Wpływ temperatury wyżarzania na mikrotwardość powłoki na bazie Ti_3SiC_2/TiC

Figure 8 shows the XRD patterns of coating based on Ti_3SiC_2 before and after annealing. The results of X-ray phase analysis of coatings showed that the coating before annealing consists of phases TiC and Ti_3SiC_2.

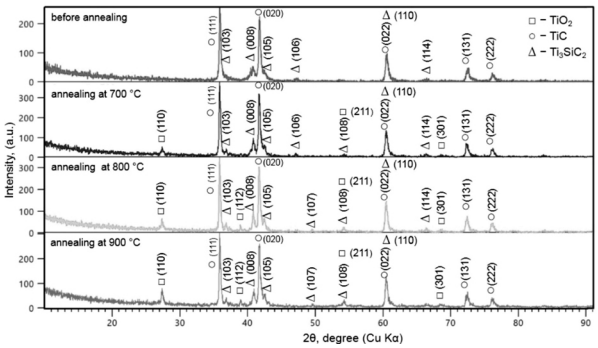


Fig. 8. XRD patterns of coating based on Ti_3SiC_2 under different annealing temperature

Rys. 8. Wzory XRD powłoki na bazie Ti_3SiC_2 w różnych temperaturach wyżarzania

After annealing, the formation of TiO_2 phases and an increase in the intensity of the (103) and (108) reflections of the Ti_3SiC_2 phases are observed. After annealing at T = 900°C, a decrease in the intensity of the TiC line and an increase in the intensity of the TiO_2 line are observed, which indicates an increase in the thickness of the oxide layer. An increase in the microhardness after annealing is associated with an increase in the content of the Ti_3SiC_2 phases in the coatings. At the same time, after annealing at T = 900°C, the increase in microhardness is insignificant due to the increase

in the thickness of the oxide layer. Thus, materials based on a Ti_3SiC_2/TiC compound with a nanolaminate structure combine the properties of ceramics and metals are characterized by high values of elastic modulus (326 GPa) and shear (135 GPa), significant fracture toughness (7–12 MPa·$m^{0.5}$), strength, crack resistance, heat resistance, chemical resistance, and low density (4.52 g/cm^3) **[L. 4–7, 16].**

The results of tribological experiments of coatings showed that the temperature of thermal annealing and the structure of the coatings themselves have a significant impact on the value of the coefficient of friction of the surface of coatings and wear resistance. Therefore, in the case of composite coatings Ti_3SiC_2/TiC before annealing, the friction coefficient is 0.65–0.70. After thermal impact at temperatures up to 800°C, the coefficient of friction at the initial stage of testing (up to 12.40 m) is 0.30–0.35 and there is a slight increase in which the coefficient of friction monotonically increases from 0.35 to 0.70 as in the case before annealing **(Fig. 9)**. This is apparently due to the fact that the coating has a higher wear resistance than the substrate. According to the result of X-ray phase analysis, an increase in the wear resistance of the near-surface layers of the Ti_3SiC_2/TiC composite material after 800°C is associated with the formation of TiO_2 **(Fig. 8)**. In **[L. 18–20]**, it is stated that the oxide compound based on TiO_2 increases the wear resistance and strength of materials.

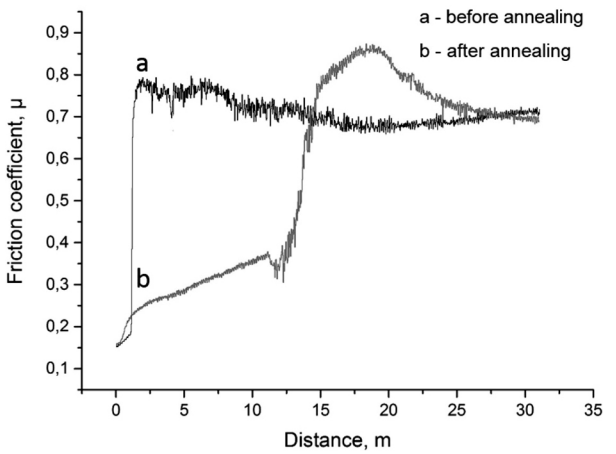


Fig. 9. Results of tribological experiments of composite coatings Ti_3SiC_2/TiC before and after annealing

Rys. 9. Wyniki badań tribologicznych powłok kompozytowych Ti_3SiC_2/TiC przed i po wyżarzaniu

With the help of profilometer, pictures of the wear track of the test samples were taken before and after annealing **(Fig. 10)**. When assessing the wear resistance of the samples based on the geometrical parameters of the wear tracks, it can be said that the depth of the sample after annealing is much smaller compared to the sample before annealing.

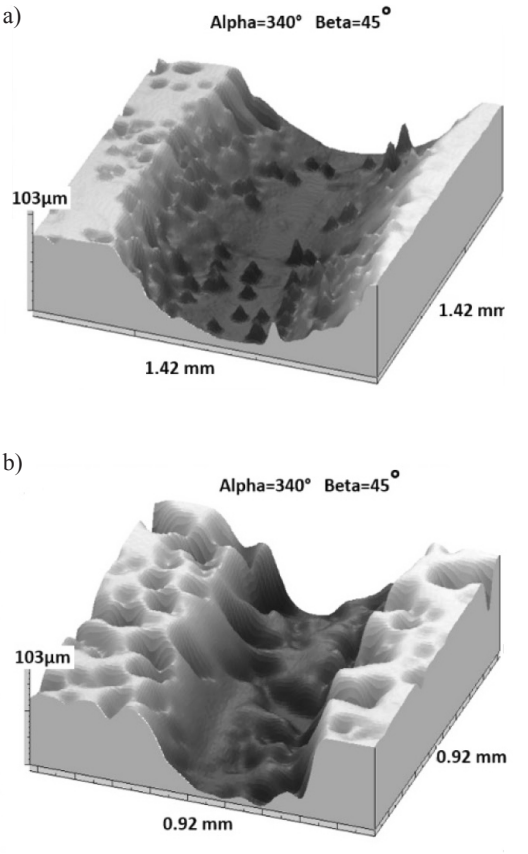


Fig. 10. Track profiles of Ti_3SiC_2/TiC composite coatings: (a) before annealing, (b) after annealing

Rys. 10. Profile śladów kompozytowych powłok Ti_3SiC_2/TiC: (a) przed wyżarzaniem, (b) po wyżarzaniu

The wear rate under the influence of the tip is calculated based on the volume of the displaced material during the test, which was calculated by the following **Formula 1:**

$$I = \frac{V}{F \times l} \tag{1}$$

where I – wear rate, [mm^3/N*m]; l is the friction path, [m]; F– nominal load, [N]; V is the volume of the worn part, [mm^3].

As a result of the calculations, we obtained the data on the wear rate for the samples before and after annealing the composite coatings Ti_3SiC_2/TiC, which are given in **Table 2**.

Table 2. Data on wear rate and wear volume of composite coatings Ti_3SiC_2/TiC

Tabela 2. Dane dotyczące szybkości zużycia i objętości zużycia powłok kompozytowych Ti_3SiC_2/TiC

№	Name of samples	Intensity of wear, μm^3	Wear volume, μm^3
1	before annealing	8450 ±1098	1763.6 ±229
2	after annealing	1498 ±194	154.1 ±20

CONCLUSIONS

Thus, the detonation method allows one to obtain coatings based on carbosilicide on the surface of steel U9/У9. The microstructure of the coating has a layered structure. The border between the coating and the base has a characteristic crinkled appearance. The Ti_3SiC_2 coating is characterized by increased microhardness (700 $HV_{0.2}$) compared to the substrate (250 $HV_{0.2}$). It was determined that the phase composition of the coatings changes during detonation spraying as a result of the decomposition of Ti_3SiC_2 powder into titanium carbide, due to the high-speed impact interaction heated to high temperatures. Thus, the selected consolidation conditions ensure the formation of the Ti_3SiC_2/TiC composite material. The presence in the composition of the Ti_3SiC_2 coating of a larger proportion of the hardening carbide phase of TiC indicates an increased resistance to abrasive wear. It was found that the maximum microhardness of the Ti_3SiC_2/TiC composite material after annealing at a temperature of 800°C is explained by an increase in the content of the Ti_3SiC_2 phase. It is possible that a decrease in microhardness at a temperature of 900°C and an increase in the wear resistance of the near-surface layers of the $T_{i3}SiC_2$/TiC composite material are associated with the formation of TiO_2 after annealing. It was established that, during detonation spraying of Ti_3SiC_2 powders, a coating with a higher microhardness and wear resistance is formed.

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