

Zh.B. Sagdoldina¹, B.K. Rakhadilov^{1,2}, D.B. Buitkenov^{1,*}, L.G. Zhurerova¹, A.B. Kenesbekov³

¹*S. Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan*

²*Plasma Science LLP, Kazakhstan*

³*Institute for Composite Materials, Ust-Kamenogorsk, Kazakhstan*

(*E-mail: buitkenovd@mail.ru)

Improvement of tribological properties of detonation carbosilicide coatings with subsequent pulsed-plasma treatment

This work considers the results of research of mechanical and tribological properties of surface layer of Ti_3SiC_2 coatings after exposure to pulsed-plasma energy flows. Varying of technological parameters of pulsed-plasma treatment is made by changing the distance of pulse exposure. The analysis of the obtained results shows that the pulsed-plasma treatment technology makes it possible to improve the properties of Ti-Si-C based coatings by strengthening the deposited compositions by modifying the structure with an increase in the number of MAX-phases. It is established that the modification of the structural and phase state of the near-surface layers of the carbosilicide coatings leads to a change in their mechanical properties: an increase in the surface microhardness up to 1.8 times, a decrease in the dry friction coefficient by 1.5-2.0 times and an increase in wear resistance by 2.5 times. Based on the XRD analysis, it is established that the improvement of mechanical and tribological properties of Ti_3SiC_2 detonation coatings as a result of pulsed-plasma treatment is associated with phase transformations in the surface layer, in particular with an increase in the Ti_3SiC_2 phase content.

Keywords: detonation spraying, pulsed-plasma modification, tribology, phase, microhardness, wear resistance.

Introduction

The structure and properties of Ti-Si-C-based detonation coatings can be regulated with subsequent heat treatment. The content of Ti_3SiC_2 phases in the coatings can be increased (restored) to some extent depending on the thermal annealing temperature. The results given in papers [1–4] confirm that increasing the volume fraction of Ti_3SiC_2 provides high mechanical and tribological properties of coatings. Thermal stabilization minimizes the residual deformation and residual stresses but has some disadvantages. For example, heat treatment has a considerable time duration and is energy intensive. In addition, there is the need for design and fabrication of accompanying tooling, as well as high capital costs for the purchase and installation of large furnaces [5]. There are also disadvantages associated with the weakening of the substrate material.

Nowadays, methods of surface treatment of products using combined processing technologies are being intensively developed [6, 7]. Surface modification can be performed by various methods, including machining with concentrated energy fluxes. Literature analysis of high-energy density treatment methods used for surface modification of parts shows that they provide multiple increases in the productivity of parts operating in a wide variety of conditions. Among them, of particular interest is the use of pulsed plasma treatment methods [8, 9], which are not inferior, and sometimes even superior, to laser, electron-beam, electric discharge, and other treatment methods. Pulsed plasma treatment is a high-performance surface modification process that is carried out without heating the entire product. It makes it possible to solve the problems of increasing the wear resistance of a particular surface without changing the structural state of the whole product [10]. The advantage is also the possibility of a local impact on the product by pulsed plasma [11].

In this work, the results obtained for the first time on the effect of pulsed plasma treatment (PPT) on the mechanical and tribological properties of Ti-Si-C-based coatings are presented.

Experimental

Detonation spraying was carried out using the CCDS2000 (Computer-Controlled Detonation Spraying) installation developed at the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia. The design and advantages of the barrel of this geometry are described in work [12, 13]. The coatings were obtained at a ratio of $O_2/C_2H_2 = 1.856$, with explosive mixture volumes of 60% and using nitrogen as a carrier

gas. The coatings were applied at a detonation gun firing rate of 2 rounds per second. The coatings were applied to U9 low-carbon steel substrates at a distance of 200 mm. Before spraying, the substrates were sand-blasted for better adhesion of the coatings.

Surface modification of the coatings was carried out by pulsed plasma flow using a plasma generator "Pulse 6" developed by the E.O. Paton Electric Welding Institute at the National Academy of Sciences (NAS) of Ukraine [14, 15]. The detonation coatings were processed under the following conditions: capacitance of capacitors 960 μF , voltage 3,2 kV, inductance 3×10^{-2} mH, electrode W, frequency 1.2 Hz, speed of passage 5 mm/s, electrode recess $h=16$ mm, number of passes 1. During varying the technological parameters of the pulsed plasma treatment, the distance of the impacts with pulses was changed from 30 to 50 mm.

X'PertPro (Philips Corporation, Nederland) using $\text{CuK}\alpha$ radiation was used for X-ray studies of coatings. The shooting was carried out in the following modes: tube voltage $U=40$ kV; tube current $I=20$ mA; exposure time 1 s; shooting step 0.02° . The microhardness from the coating's surface was measured using the Metolab-502 testing machine (Metolab, Russia) according to GOST 9450-76. The load on the Vickers pyramid was 200 g. Tribological tests of coatings were carried out on a TRB³ tribometer (Anton Paar Srl, Peseux, Switzerland) according to the "ball-disc" scheme based on ASTM G-99. An aluminium oxide ball with a diameter of 6 mm was used as a counter body. The samples were tested at a normal load of 10 N, a wear radius of 5 mm, a sliding speed of 2 m/s^{-1} and a total sliding distance of 100 m. The CSEM Micro Scratch Tester (Neuchatel, Switzerland) was used to study the adhesive characteristics of coatings by the "scratching" method. Scratch testing was performed at a maximum load of 30 N; the rate of change of normal loading on the sample was 29.99 N/min, the speed of movement of the indenter was 9.63 mm/min, the length of the scratch was 10 mm, the radius of tip curvature was 100 microns. To obtain reliable results, three scratches were applied to the surface of each coated sample. The roughness (Ra) of the coating surface was measured using a profilometer model 130 (JSC Plant PROTON, Russia). Coatings testing for abrasive wear are carried out on an abrasive-erosion stand by ASTM G65 standards, the principles of which are similar to GOST 23.208-79. In abrasive tests of rubber coatings, a disk with a diameter of 50 mm rotating at a speed of 60 rot/min rubs against a stationary sample. In addition, dry abrasive material is fed to the friction surface. The sample is pressed against the disk with a force of 130 N; electrocorundum powder with sharp-angled particles of 150...190 microns in size is fed to the contact surface. The sample testing procedure consists of 3-5 tests, each of which lasts 15 minutes (600 disk revolutions). After each test, the sample is weighed on an analytical balance. The volume wear is considered, which is determined by dividing the mass loss by the density of the coating material. In erosion tests, a stream of abrasive particles is applied to the surface of the sample at a given angle (30° or 90°) by an air jet. The same sand is used as in abrasive tests. Sand consumption is 1.3g/min, particle velocity is 60m/s, air consumption is $0.12 \text{ m}^3/\text{min}$. 8 tests are done, each lasting 5 minutes. Mass loss is recorded; the results are recorded as a mass loss in 5 minutes. Further, as well as abrasive tests, the volume loss is determined in 5 minutes.

Results and Discussion

Figure 1 presents diffractograms of Ti-Si-C system coatings before and after different pulsed plasma treatment distances. The results of the X-ray phase analysis of the coatings show that the phase composition of the coatings before the PPT consists mainly of TiC and a relatively small fraction of Ti_3SiC_2 . After PPT, an increase in the intensity of Ti_3SiC_2 peaks is observed as well as the appearance of new reflexes (101, 102, 112, 204, 1110, 0016) of this phase which indicates an increase in the MAX-phase content. The change in phase fraction designates a solid-phase transformation during pulsed plasma activation associated with heating above the melting temperature and cooling of the samples during treatment [16, 17]. The cooling rate of the sample and the crystallization rate of the melt (processed layer) depend on the heat capacity of the base metal (substrate). In diffractograms of coatings, the carbide and oxide phases: WC and TiO_2 are present in small amounts. The samples were treated in an air environment, which caused the formation of oxide phases. Tungsten carbide is formed due to the consumption of the tungsten electrode [18].

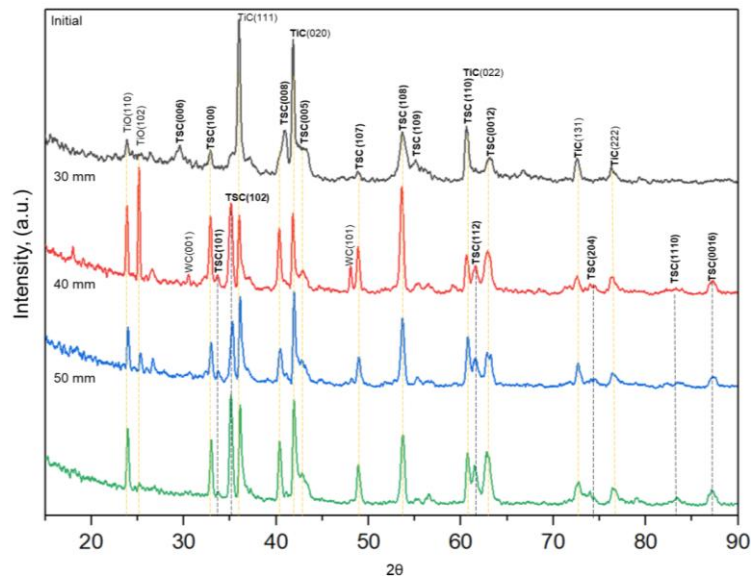


Figure 1. Diffractograms of Ti-Si-C-based detonation coatings before and after PPT

Microhardness of coatings after pulsed plasma treatment increases in comparison with an initial sample in dependence on the distance of treatment (Table 1). The values of microhardness of coatings after PPT application at a distance of 50 mm increased up to ~1785 HV (before PPT ~1000 HV) due to more effective formation of MAX phases.

To assess the resistance of Ti-Si-C coatings to abrasive and erosive wear, tests were carried out on special stands. Comparative studies of the coatings' wear resistance under friction in an abrasive medium showed that after modification by plasma treatment, coatings provide the greatest wear resistance. Table 1 represents the results of testing for abrasive and erosive wear of coatings before and after pulsed plasma treatment depending on the distance from the plasmatron. According to the results of determining the mass losses of the samples after the test, the maximum resistance to all types of wear is provided by the coating treated at a distance of 50 mm.

Table 1

Abrasive and erosive wear of detonation coatings before and after PPT

Coating	Hardness [HV]	Abrasive wear [mg]	Erosive wear [mg]
Ti ₃ SiC ₂	1000	0.87	0.38
PPT (30 mm)	1180	0.65	0.29
PPT (40 mm)	1250	0.59	0.25
PPT (50 mm)	1785	0.52	0.23

An important characteristic of the parts working surfaces is the friction coefficient and wear resistance. Tribological properties are determined by the structural-phase state, strength, and surface chemical properties. Figure 2 demonstrates the curves of wear of coated Ti-Si-C before and after modification by plasma treatment, depending on the distance from the plasmatron. The experiment shows that after the pulsed-plasma treatment of the samples, the friction coefficient μ decreases. If the value of the friction coefficient in the initial Ti-Si-C coatings is 0.65, then after the pulse treatment it decreases depending on the distance H from 0.60 up to 0.40. According to the experiments, pulsed plasma treatment leads to an improvement in the tribological properties of Ti-Si-C coatings. A possible reason for the decrease in the friction coefficient is an increase in the content of the Ti₃SiC₂ phase on the surface layer of coatings after pulsed plasma treatment.

The analysis of the surface roughness shows that the value of the arithmetic mean deviation of the initial coating roughness profile was 0.97 μm (Fig. 2). After pulsed-plasma treatment, this value increased at a distance of 30 mm to 3.75 μm , at 40 mm to 3.61 μm and at 50 mm to 3.53 μm . An increase in the surface roughness compared to the initial sample leads to a decrease in the actual contact area of the interacting bodies, which also causes a decrease in the friction coefficient [19].

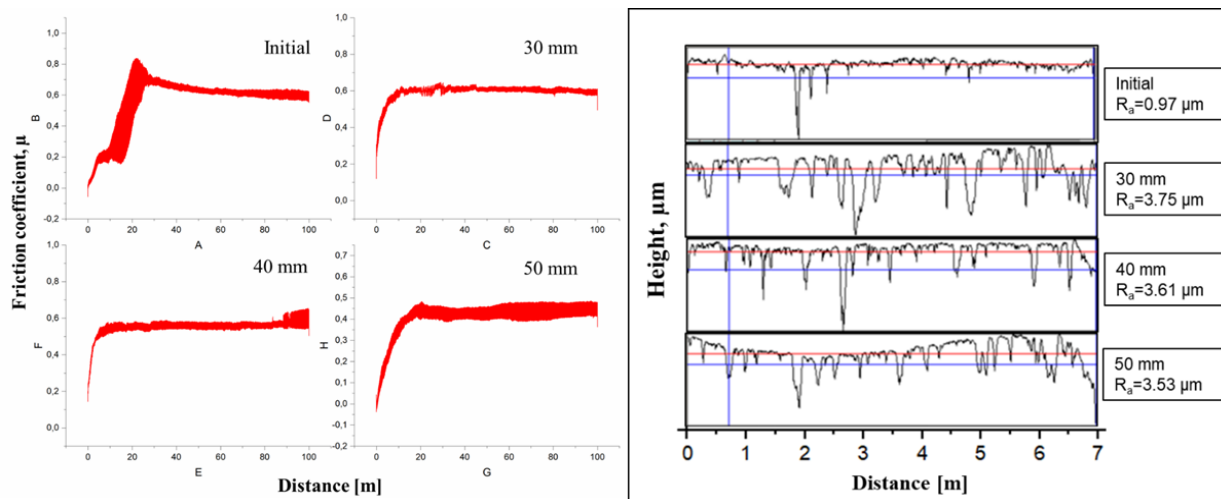


Figure 2. Results of tribological tests of Ti-Si-C coatings before (initial) and after pulsed-plasma treatment

Figure 3 illustrates the results of adhesion and cohesion strength and scratch resistance of Ti-Si-C coatings before and after the PPT. The process of coating failure by indenter scratching can be conventionally divided into three stages. Coatings before PPT at a load in the range of 12.08 N (L_{C1}) show monotonic penetration of the indenter into the coating. At a load of 24,38 N (L_{C2}) the indenter fully sinks into the coating. A sliding diamond indenter to perform a coating with a coefficient of friction of 0.35. As the load is increased by 28.92 N (L_{C3}), the material in front of the indenter is squeezed into knolls and the penetration depth of the indenter increases. Comparative analysis shows that the coatings after PPT erode but do not delaminate when scratched, i.e., they fracture due to the cohesive mechanism of plastic deformation and the formation of fatigue cracks in the coating material. As we can see, there is a monotonic penetration of the indenter into the coating and the first cracks appear (load up to 18.02 N); the coefficient of friction (μ) increases, but the acoustic emission signal remains unchanged. Subsequently, chevron and diagonal cracks appear at increased load, which increases the coefficient of friction to a value of 0.3. Under load up to 18-25 N, the amplitude of the acoustic emission signal increases sharply. Thereafter, with an increase in load reaching 29.88 N, local abrasion of the coating down to the substrate material occurs.

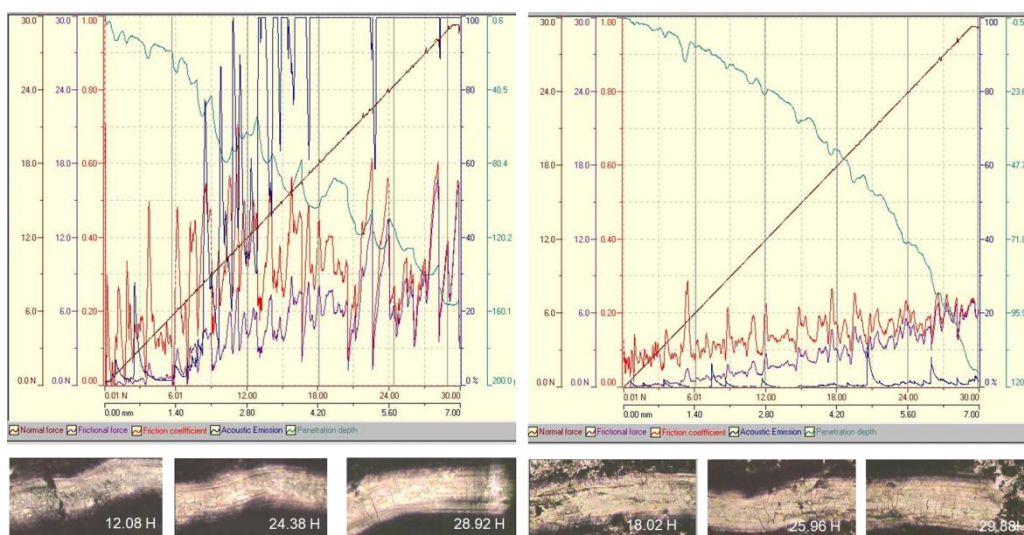


Figure 3. Scratch test results of Ti_3SiC_2 coatings before and after PPT

Conclusions

It is determined that after PPT the intensity of Ti_3SiC_2 peaks increases and new reflexes appear (101, 102, 112, 204, 1110, 0016) which indicates an increase of MAX phase content. Formation of carbide and oxide phases (WC and TiO_2) in small amounts is connected with the evaporation of tungsten electrodes dur-

ing PPT in air environment. It is shown that before PPT the average coefficient of friction of coatings is ~ 0.60 , after treatment the coefficient of friction decreases and is from 0.55 to 0.40 depending on treatment distance. The reason for the friction coefficient reduction may be an increase in microhardness and an increase in the content of MAX phases in the composition of the coatings. After PPT at the distance of 50 mm, the wear resistance of coatings to abrasive and erosive wear increases by 1.5–2.0 times. Thus, when pulsed plasma is treated with detonation coatings based on Ti-Si-C, a modified layer appears on the surface, which, in terms of its mechanical and tribological properties, is superior to the original surface.

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Ж.Б. Сағдолдина, Б.К. Рахадиллов, Д.Б. Буйткенов, Л.Г. Журерова, А.Б. Кенесбеков

Импульстік–плазмалық өңдеумен детонациялық карбосилицидтік жабындардың трибологиялық қасиеттерін жақсарту

Мақалада импульстік-плазмалық энергия ағындарының әсерінен кейін Ti₃SiC₂ жабындарының беткі қабатының механикалық-трибологиялық қасиеттерін зерттеу нәтижелері қарастырылған. Импульстік-плазмалық өңдеудің технологиялық параметрлерінің түрленуі импульстердің әсер ету қашықтығының өзгеруіне байланысты жүргізілді. Алынған нәтижелерді талдау импульстік-плазмалық өңдеу технологиясы МАХ-фазалар санын ұлғайта отырып, құрылымды түрлендіру жолымен қолданылған композицияларды нығайта отырып, Ti-Si-C жүйесі негізінде жабындардың қасиеттерін жақсартуға мүмкіндік беретінін куәландырады. Карбосилицидті жабындардың беткі қабатының құрылымдық-фазалық күйін өзгерту олардың механикалық сипаттамаларының өзгеруіне әкелетіні анықталды: беттің микро қаттылығын 1,8 есеге дейін арттыру, құрғақ үйкеліс коэффициентін 1,5–2,0 есе азайту және тозуға төзімділікті 2,5 есе арттыру. XRD талдау негізінде импульстік плазмалық өңдеу нәтижесінде Ti₃SiC₂ детонациялық жабындарының механикалық-трибологиялық қасиеттерінің жақсаруы беткі қабатындағы фазалық өзгерістермен, атап айтқанда Ti₃SiC₂ фазасының ұлғаюымен байланысты екендігі анықталды.

Кілт сөздер: детонациялық тозаңдату, импульсті-плазмалық модификация, трибология, фаза, микроқаттылық, тозуға төзімділік.

Ж.Б. Сағдолдина, Б.К. Рахадиллов, Д. Б. Буйткенов, Л.Г. Журерова, А.Б. Кенесбеков

Повышение трибологических свойств детонационных карбосилицидных покрытий с последующей импульсно-плазменной обработкой

В статье рассмотрены результаты исследования механико-трибологических свойств поверхностного слоя покрытий Ti₃SiC₂ после воздействия импульсно-плазменными потоками энергии. Варьирование технологических параметров импульсно-плазменной обработки производилось за счет изменения дистанции воздействия импульсами. Анализ полученных результатов свидетельствует о том, что технология импульсно-плазменной обработки позволяет улучшать свойства покрытий на основе системы Ti-Si-C, упрочняя нанесенные композиции путем модифицирования структуры с увеличением количества МАХ-фаз. Установлено, что модифицирование структурно-фазового состояния приповерхностных слоев карбосилицидных покрытий приводит к изменению их механических характеристик: увеличению микротвердости поверхности до 1,8 раз, уменьшению коэффициента сухого трения в 1,5–2,0 раза и повышению износостойкости на 2,5 раза. На основании XRD-анализа установлено, что улучшение механико-трибологических свойств детонационных покрытий Ti₃SiC₂ в результате импульсно-плазменной обработки связано с фазовыми превращениями в поверхностном слое, в частности, с увеличением содержания фазы Ti₃SiC₂.

Ключевые слова: детонационное напыление, импульсно-плазменная модификация, трибология, фаза, микротвердость, износостойкость.

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