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## Influence of pulse plasma treatment on the phase composition and microhardness of detonation coatings based on Ti-Si-C

The paper considers the study results of the phase composition and microhardness of detonation coatings based on Ti-Si-C after exposure to pulsed plasma treatment. The CCDS2000 detonation complex was used to obtain the coatings. Coatings surface modification was carried out using pulsed plasma exposure (PPE). The detonation coatings were treated with varying the distance H (30 mm mode 1, 40 mm mode 2, 50 mm mode 3) from the plasmatron to the hardened surface. It is shown that the treated coatings are generally characterized by high microhardness compared to the original coating. It was determined that after treatment by pulsed plasma effects an increase in the intensity of all reflexes phase  $Ti_3SiC_2$  was observed, and the appearance of reflexes (101, 102, 112, 204, 0016) phase  $Ti_3SiC_2$  was found, which indicates the increase of the content of  $Ti_3SiC_2$  phase. The change in the fraction of phases indicates a solid-phase transformation during pulsed-plasma activation. High hardness is observed on the coating surface treated according to mode 3 (50 mm) and as it approaches the surface of the substrate modes 2 (40 mm) and 1 (30 mm) the hardness decreases. It is established that the increase in the microhardness of detonation coatings after pulse-plasma treatment is associated with an increase in the content of  $Ti_3SiC_2$  phases in the coatings, as well as an increase in the defects density in the modified layer.

*Keywords:* detonation spraying, pulse plasma treatment, phase, microhardness, modification, carburized titanium, steel U9, coating.

### Introduction

The current level of development of the theory and practice of increasing the metal's hardness and wear resistance requires the creation of protective layers with high mechanical performance. In many industries coatings made of refractory powder materials (for example, Ti-Si-C) deposited by gas-thermal methods (plasma, detonation) are widely used [1, 2]. They have considerable interest due to their unique combination of metallic and ceramic properties. As metals, they have good electrical and thermal conductivity, high plasticity, good machinability and excellent resistance to thermal shock. As ceramics, they have low density, high stiffness, high melting points, and good resistance to oxidation and corrosion [3, 4]. Such exceptional properties arise from the coexistence of strong covalent-ionic MX bonds and weak metallic MA bonds within the layered hexagonal structure (space group P63/mmc) of MAX materials, which are created by repeating a three-layer structure (consisting of two layers of Mn + 1xN intercalated by a single atomic layer A) [5]. However, obtaining of coatings based on Ti-Si-C by traditional methods is associated with a high temperature and the long duration of the process. The spraying of coatings based on  $Ti_3SiC_2$  is usually accompanied by the formation of Ti-C and Ti-Si phases. The short reaction time of powder mixtures and the decomposition of Ti-Si-C at high temperature are the main problems for the purity of the phase [6]. Expected hard working conditions put forward a number of requirements for the morphology and structure of protective layer. The disadvantages of traditional methods can be surmounted by thermal activation of the surface by pulsed plasma flows, as well as by electron beams [7, 8]. The advantages of pulse-plasma technology are high rates of heating and cooling of the material surface ( $10^4$ - $10^8$  Ks<sup>-1</sup>), the high deposition rate of coatings and low reagent consumption, wide variation in the parameters of the plasma flow (number of pulses, specific power), the possibility of creating layered structures with different phase composition and, accordingly, with different physical and chemical characteristics [9, 10]. The possibility of local exposure to the product with pulsed plasma is also advantageous [11, 12]. Thus, the pulse-plasma technology allows flexible control of the surface layer modifying process.

The aim of this work is to study the effect of pulsed plasma treatment on the phase composition and microhardness of detonation coatings based on Ti-Si-C.

### *Material and methods of research*

Detonation spraying method was used to obtain coatings based on  $Ti_3SiC_2$  on the U9 steel surface (with 0.94 wt % C). Powder chemical composition: Ti – 74 wt.%; SiC – 20 wt.%; C – 6,0 wt.%. The powder particle size ranges from 20 to 40  $\mu m$ . Before applying the coatings sandblasting was carried out in aim to improve the coating's adhesive strength.

The CCDS2000 detonation complex, which has a system of electromagnetic gas valves that regulate the supply of fuel and oxygen, as well as control the purging of the system, was used to obtain the coatings [13]. The CCDS2000 allows applying coatings from a wide range of materials to various substrates. The main elements of the complex are shown in Figures 1 a, b, c. The working body (gun) can also be placed on an industrial robot (Figure 1c).



Figure 1. Detonation complex CCDS2000

- (a) working body (gun) consisting of a barrel, a gas distribution unit and powder dispensers; (b) the control unit on the basis of the industrial computer; (c) the gun is complete with a 3-coordinate manipulator

Detonation spraying process is schematically shown in Figure 2.

Gas acetylene-oxygen mixture, the most popular fuel for detonation spraying of powder materials, was used as a fuel. The spraying was carried out at the  $O_2/C_2H_2 = 1.856$  ratio of the acetylene-oxygen mixture. Explosive gas mixture volume of the detonation gun barrel is 60%. Nitrogen was used as a carrier gas. The distance between the sample treated surface and the detonation barrel was 200 mm. The diameter of the straight barrel was 20 mm.

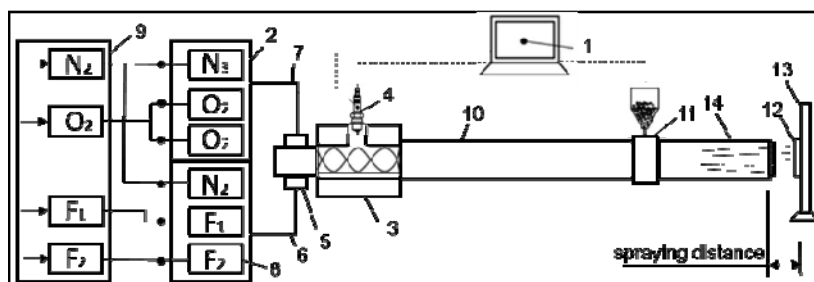


Figure 2. Principled schematic diagram of the CCDS2000 detonation complex.

- 1 – control computer, 2 – gas distributor, 3 – mixing-ignition chamber, 4 – spark plug, 5 – barrel valve, 6 – fuel line, 7 – oxygen line, 8 – gas valves, 9 – gas supply unit, 10 – indicated part of the barrel, 11 – powder dispenser, 12 – workpiece; 13 – manipulator, 14 – the muzzle of the barrel

Surface modification of the coatings was carried out by pulsed plasma flow using a plasma generator developed by the E. O. Paton Institute of Electric Welding at NAS of Ukraine [14, 15]. The features of which is the possibility of commuting the electric current by the ionized region of the gas behind the detonation wave front. This makes it possible to generate pulsed plasma with a frequency of 1-4 Hz and energy of up to 7 kJ. The pulse-plasma generator (Fig. 3) consists of a detonation chamber (1), where the combustion of combustible gas mixture ( $C_3H_8$ ,  $O_2$ , air) is initiated, and coaxial electrodes (2, 3). If it is necessary to introduce alloying elements into the plasma in the form of vapour drop phase an erodible electrode (4) is used.

When an electric current flows behind the detonation wave (5), Joule heat is released in the interelectrode gap, thereby enhancing the accelerating plasma force's electromagnetic and gas-dynamic components. The energy characteristics of the plasma jets at the output of the pulsed plasma device depend on the geometry of the coaxial electrodes and the electric field strength in the interelectrode gap. When the electric field strength between the electrodes is  $3.5 \times 10^5$  V/m, the plasma velocity at the exit from the plasma torch reaches 4 km/s, and the temperature is 12000 K. At the moment of the interaction of the plasma pulse with the surface of the product (6), a region of shock-compressed plasma layer is formed in the contact zone. The electrical circuit between the eroded electrode and the product closes up. The current density reaches  $10000 \text{ A/cm}^2$ . As a result, the surface undergoes multiple impacts, including thermal with a power density of  $104 \dots 106 \text{ W/cm}^2$ , chemical, electromagnetic (magnetic field strength up to  $4 \times 10^5 \text{ A/m}$ ). Repeated exposure (thermal cycling) allows changing the surface's physical and mechanical properties due to structural transformations [15].

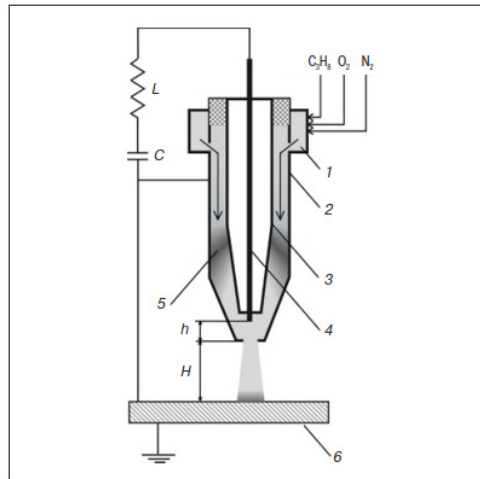


Figure 3. Pulsed plasma device scheme [16]

1 – detonation chamber; 2, 3 – coaxial electrodes; 4 – consumable electrode; 5 – detonation wave; 6 – treating part

The detonation coatings were treated with a pulsed plasma jet under the following modes: capacitance of capacitors  $960 \mu\text{F}$ , voltage  $3,2 \text{ kV}$ , inductance  $3 \times 10^{-2} \text{ mH}$ , electrode  $W$ , frequency  $1.2 \text{ Hz}$ , speed of passage  $5 \text{ mm/s}$ , electrode recess  $h=16 \text{ mm}$ , number of passes 1.

The treatment modes were changed by varying the distance  $H$  (Fig. 3) from the plasmatron to the hardened surface (Table 1).

Table 1

Sample treatment modes

Number of hardening mode	Distance from plasma torch to product, mm
1	30
2	40
3	50

The research phase composition of the samples was studied by X-ray diffractometer X'PertPro (Philips Corporation, Nederland) using  $\text{CuK}\alpha$  radiation. The shooting was carried out in the following modes: tube voltage  $U = 40 \text{ kV}$ ; tube current  $I = 20 \text{ mA}$ ; exposure time  $1 \text{ s}$ ; shooting step  $\Delta 2\theta \sim 0.02^\circ$  and  $2\theta = 10-90^\circ$ . The microhardness of the samples was measured by a diamond indenter on a METOLAB 502 (METOLAB, Moscow) device in accordance with GOST R ISO 6507-1 at a load of  $100 \text{ g}$  and an exposure time of  $10 \text{ s}$ . At least ten measurements were carried out on each sample, the results of which were averaged [17, 18].

#### Results and discussions

Figure 4 shows the diffractograms of the initial coatings of the Ti-Si-C system and those processed by pulsed plasma exposure. The results of the coatings' XRD analysis showed that the initial coating consists of

TiC phases as the main phases and  $Ti_3SiC_2$  as the secondary phase. The phase structure has not significantly changed after the pulse-plasma treatment, but the peaks' relative intensity considerably changed. After treatment by pulsed plasma effects an increase in the intensity of all reflexes phase  $Ti_3SiC_2$  was observed, and the appearance of reflexes (101, 102, 112, 204, 0016) phase  $Ti_3SiC_2$  was found, which indicates the increase of the content of  $Ti_3SiC_2$  phase. The change in the phase fraction indicates a solid-phase transformation during pulsed-plasma activation. According to mode 1 (30 mm), the coating has small amounts of carbide and oxide phases: WC, TiO. This is related to the material of the consumable electrode penetrating the hardened layer of the product. For example, after using consumable titanium electrode hardened layer is found at a depth of up to 20  $\mu m$  [19].

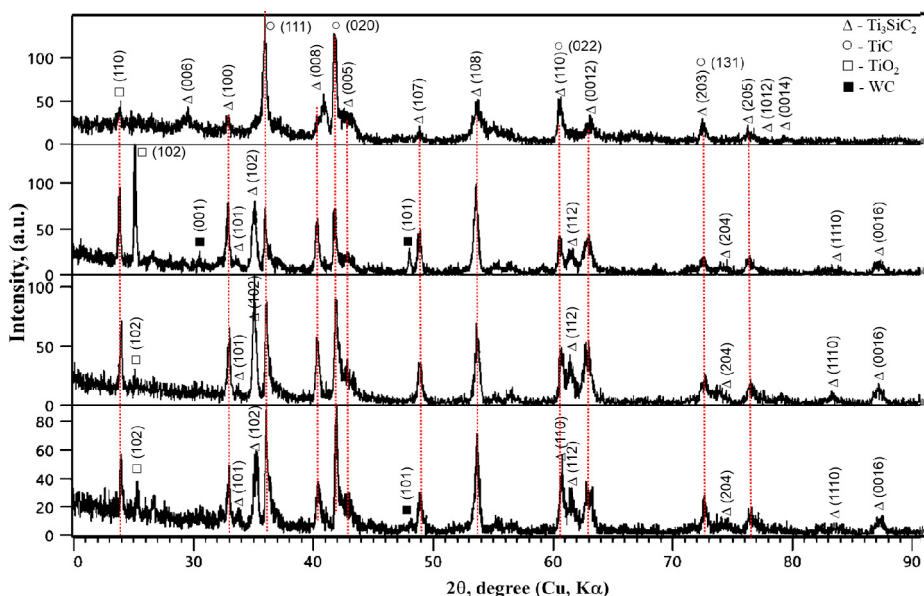


Figure 4. Diffractograms of detonation coatings based on Ti-Si-C before (a) and after pulse-plasma treatment: (b) mode 1 (30mm), (c) mode 2 (40mm), (e) mode 3 (50mm)

The Vickers hardness of Ti-Si-C detonation coatings before and after pulsed plasma exposure is shown in Figure 5. The treated coatings are generally characterized by high microhardness compared to the original coating. High hardness is observed on the coating surface treated according to mode 3 (50 mm) and as it approaches the surface of the substrate modes 2 (40 mm) and 1 (30 mm) the hardness decreases. According to the results of the XRD analysis, the increase in the detonation coatings' microhardness of the Ti-Si-C system because of pulse-plasma treatment is associated with an increase in the content of  $Ti_3SiC_2$  phases in the coatings, as well as an increase in the density of defects in the modified layer.

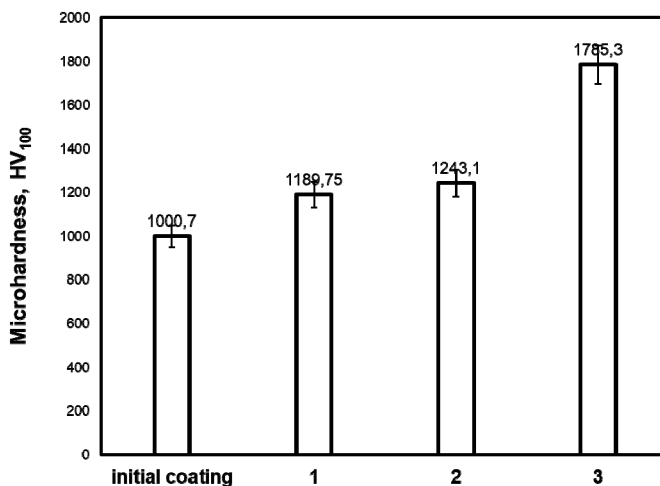


Figure 5. Microhardness of detonation coatings based on Ti-Si-C before and after pulsed plasma treatment

### Conclusions

The influence of modification by pulsed-plasma exposure to Ti–Si–C-based coatings obtained by the detonation method was studied. The study showed that after treatment by pulsed plasma effects an increase in the intensity of all reflexes phase  $Ti_3SiC_2$  was observed, and the appearance of reflexes (101, 102, 112, 204, 0016) phase  $Ti_3SiC_2$  was found, which indicates the increase of the content of  $Ti_3SiC_2$  phase. The change in the phase fraction indicates a solid-phase transformation during pulsed-plasma activation. The treated coatings are generally characterized by high microhardness compared to the original coating. According to the results of XRD analysis, the increase in the microhardness of the detonation coatings of the Ti-Si-C system because of pulse-plasma treatment is associated with an increase in the content of  $Ti_3SiC_2$  phases in the coatings, as well as an increase in the density of defects in the modified layer.

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## Импульстік-плазмалық өңдеудің Ti–Si–C негізіндегі детонациялық жабындардың фазалық құрамына және микроқаттылығына әсері

Мақалада импульстік плазмалық өңдеу әсерінен кейінгі Ti–Si–C негізіндегі детонациялық жабындардың фазалық құрамын және микроқаттылығын зерттеу нәтижелері қарастырылды. Жабындарды алу үшін CCDS-2000 детонациялық кешені қолданылды. Жабындарды беттік модификациялау импульстік-плазмалық әсерді (ИПӨ) қолдану арқылы жүзеге асырылды. Детонациялық жабындарды өңдеу плазматроннан қатайтылатын бетке дейін әртүрлі арақашықтықты (30 мм – 1; 40 мм – 2; 50 мм – 3 режим) пайдалана отырып жүзеге асырылды. Өңделген жабындар, әдетте, бастапқы жабынмен салыстырғанда жоғары микроқаттылықпен сипатталатындығы көрсетілген. Импульстік плазмалық әсермен өңдегеннен кейін  $Ti_3SiC_2$  фазасының барлық рефлексстерінің қарқындылығының жоғарылауы, сонымен қатар  $Ti_3SiC_2$  фазасының (101, 102, 112, 204, 0016) рефлексстерінің пайда болуы анықталды, бұл  $Ti_3SiC_2$  фазасының көбеюін көрсетеді. Фазалық бөліктің өзгеруі импульстік плазмалық активтендіру кезінде қатты фазалық түрленуді білдіреді. Жоғары қаттылық 3-ші (50 мм) режиммен өңделген жабын бетінде байқалады және субстрат бетіне жақындаған сайын 2-ші (40 мм) және 1-ші (30 мм) режимдер қаттылығы азаяды. Импульстік-плазмалық өңдеуден кейін детонациялық жабындардың микроқаттылығының артуы жабындардағы  $Ti_3SiC_2$  фазалары құрамының артуымен, сондай-ақ түрлендірілген қабаттағы ақаулардың тығыздығымен байланысты екені анықталды.

*Кілт сөздер:* детонациялық тозаңдату, импульстік-плазмалық өңдеу, фаза, микроқаттылық, модификация, титан карбосилициді, болат У9, жабын.

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## Влияние импульсно-плазменной обработки на фазовый состав и микротвёрдость детонационных покрытий на основе Ti–Si–C

В статье рассмотрены результаты исследования фазового состава и микротвёрдости детонационных покрытий на основе Ti–Si–C после воздействия импульсно-плазменной обработкой. Для получения покрытий использован детонационный комплекс CCDS–2000. Поверхностное модифицирование покрытий осуществлялось с применением импульсно-плазменного воздействия. Обработка детонационных покрытий производилась с использованием различной дистанции (30 мм – режим 1; 40 мм – 2; 50 мм – режим 3) от плазматрона до упрочняемой поверхности. Показано, что обработанные покрытия, в целом, характеризуются высокой микротвёрдостью по сравнению исходным покрытием. Определено, что после обработки импульсно-плазменным воздействием наблюдается увеличение интенсивности всех рефлексов фазы  $Ti_3SiC_2$ . Кроме того, обнаружено появление рефлексов (101, 102, 112, 204, 0016) фазы  $Ti_3SiC_2$ , что свидетельствует о росте содержания фазы  $Ti_3SiC_2$ . Изменение доли фаз указывает на твердофазное превращение при импульсно-плазменной активации. Высокая твёрдость наблюдается на поверхности покрытия, обработанной по режиму 3 (50 мм), и по мере приближения к поверхности подложки (режимы 2 (40 мм) и 1 (30 мм)) твёрдость уменьшается. Установлено, что повышение микротвёрдости детонационных покрытий после импульсно-плазменной обработки связано с увеличением содержания фаз  $Ti_3SiC_2$  в покрытиях и плотности дефектов в модифицированном слое.

*Ключевые слова:* детонационное напыление, импульсно-плазменная обработка, фаза, микротвёрдость, модификация, карбосилицид титана, сталь У 9, покрытие.

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