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STUDIES OF THE STRUCTURAL AND PHASE STATE OF DETONATION COATINGS BASED ON THE FE–TIB$_2$–CRB$_2$ EUTECTIC SYSTEM AFTER PULSE PLASMA TREATMENT

ИССЛЕДОВАНИЕ СТРУКТУРНОГО И ФАЗОВОГО СОСТОЯНИЯ ДЕТОНАЦИОННЫХ ПОКРЫТИЙ НА ОСНОВЕ ЭВТЕКТИЧЕСКОЙ СИСТЕМЫ FE–TIB$_2$–CRB$_2$ ПОСЛЕ ИМПУЛЬСНО-ПЛАЗМЕННОЙ ОБРАБОТКИ

ИМПУЛЬСТІК-ПЛАЗМАЛЫҚ ӨҢДЕУДЕН КЕЙІН FE–TIB$_2$–CRB$_2$ ЭВТЕКТИКАЛЫҚ ЖҮЙЕСІ НЕГІЗІНДЕГІ ДЕТОНАЦИЯЛЫҚ ЖАБЫНДАРДЫҢ ҚҰРЫЛЫМДЫҚ ЖӘНЕ ФАЗАЛЫҚ КҮЙІН ЗЕРТТЕУ

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Keywords:
pulsed-plasma treatment, coatings, phase composition, structure, detonation sprayin, hardness, eutectic system, wear.

ABSTRACT

Fe–TiB$_2$–CrB$_2$ coatings obtained by detonation spraying were modified by pulsed plasma treatment (PPT) to improve their structural and operational properties. A comprehensive analysis of the phase composition, microstructure, microhardness, wear resistance, and corrosion behaviour was performed. X-ray structural analysis confirmed the preservation of the α-Fe, TiB$_2$ and CrB$_2$ phases after treatment, with a simultaneous decrease in the proportion of γ-Fe and $Cr_{23}C_6$ and a redistribution of boride phases. Electron microscope studies revealed a densification of the surface layer and a decrease in interlamellar porosity. Microhardness in the near-surface zone increased from ~15 to 17–18 GPa (by 15–20%), indicating local strengthening. Tribological tests showed a decrease in wear depth, while electrochemical studies revealed a positive shift in corrosion potential by 0.15–0.20 V and a decrease in the rate of anodic dissolution. PPT provides an improvement in mechanical and corrosion characteristics without changing the basic phase composition of the coating.

Түйінді сөздер:
импульсно-плазменная обработка, покрытия, фазовый состав, структура, детонационное напыление, твёрдость, эвтектическая система, износ.

ТҮЙІНДЕМЕ

Покрытия Fe–TiB$_2$–CrB$_2$, полученные методом детонационного напыления, были модифицированы с помощью импульсно-плазменной обработки (PPT) с целью улучшения их структурных и эксплуатационных свойств. Проведен комплексный анализ фазового состава, микроструктуры, микротвердости, износостойкости и коррозионного поведения. Рентгеноструктурный анализ подтвердил сохранение фаз α-Fe, TiB$_2$ и CrB$_2$ после обработки, при одновре-

менном снижении доли γ-Fe и $Cr_{23}C_6$, а также перераспределении боридных фаз. Исследования с использованием электронного микроскопа выявили уплотнение поверхностного слоя и уменьшение межламеллярной пористости. Микротвердость в приповерхностной зоне увеличилась примерно с 15 до 17–18 ГПа (на 15–20 %), что свидетельствует о локальном упрочнении. Трибологические испытания показали снижение глубины износа, а электрохимические исследования выявили положительное смещение коррозионного потенциала на 0,15–0,20 В и снижение скорости анодного растворения. Импульсно-плазменная обработка обеспечивает улучшение механических и коррозионных характеристик без изменения основного фазового состава покрытия.

Ключевые слова:
импульстік-плазмалық өңдеу, жабындар, фазалық құрам, құрылым, детонациялық бүрку, қаттылық, эвтектикалық жүйе, тозу.

АННОТАЦИЯ

Детонациялық бүрку әдісімен алынған Fe–TiB_2–CrB_2 жабындары олардың құрылымдық және пайдалану қасиеттерін жақсарту мақсатында импульстік-плазмалық өңдеуге (PPT) ұшыратылды. Фазалық құрамы, микроқұрылымы, микроқаттылығы, тозуға төзімділігі және коррозиялық мінез-құлқы кешенді түрде талданды. Рентгенқұрылымдық талдау өңдеуден кейін α-Fe, TiB_2 және CrB_2 фазаларының сақталғанын растады, сонымен қатар γ-Fe мен $Cr_{23}C_6$ үлесінің төмендеуі және боридтік фазалардың қайта бөлінуі анықталды. Электрондық микроскопиялық зерттеулер беттік қабаттың тығыздалғанын және қабатаралық кеуектіліктің азайғанын көрсетті. Бетке жақын аймақтағы микроқаттылық шамамен 15-тен 17–18 ГПа-ға дейін артты (15–20%-ға), бұл жергілікті беріктенуді көрсетеді. Трибологиялық сынақтар тозу тереңдігінің төмендегенін көрсетті, ал электрохимиялық зерттеулер коррозиялық потенциалдың 0,15–0,20 В-қа оң ығысқанын және анодтық еру жылдамдығының төмендегенін анықтады. Импульстік-плазмалық өңдеу жабынның негізгі фазалық құрамын өзгертпей, оның механикалық және коррозиялық сипаттамаларын жақсартуды қамтамасыз етеді.

INTRODUCTION

In modern industries—mechanical engineering, energy, metallurgy, and transport construction—the resource intensity and durability of structural components remain key factors in technological and economic efficiency (Bhushan & Ko, 2003; Shapagina & Dushik, 2024). Operating conditions for equipment are increasingly characterised by high contact pressures, increased abrasive and adhesive wear, elevated temperatures and aggressive environments, leading to a rapid deterioration in the performance of machine components and assemblies (Shtansky et al., 2005). The increasing demands placed on equipment today, characterised by the combined effects of extremely high temperatures, corrosion and wear, mean that conventional hardening methods—such as solution treatment, changes to component geometry or standard heat treatment—are insufficient to ensure durability (Kiryukhantsev-Korneev et al., 2017).

One of the most promising areas of surface engineering is the application of protective coatings using detonation gun spraying (D-Gun) (Panarin et al., 2016), which provides dense, adhesion-stable layers with minimal porosity and good microstructural bonding between the coating particles and the substrate. This technology allows the formation of coatings with high hardness and wear resistance without significant thermal impact on the substrate, which is critical for thin-walled and heat-sensitive components.

In recent years, detonation coatings based on Fe–CrB_2–TiB_2 have been considered as a hard and extremely wear-resistant alternative to traditional carbide and oxide coatings for parts

operating under conditions of abrasive wear, impact abrasion and friction (Prysyazhnyuk et al., 2025; Shtansky et al., 2005). The demand for these compositions stems from the fact that the refractory compounds TiB_2 and CrB_2 provide exceptional hardness and structural stability under high-temperature conditions. At the same time, the ductile iron base acts as a plastic matrix, which effectively fixes the dispersed strengthening phases and forms a strong framework that prevents brittle fracture. The use of iron also ensures high adhesion and the processability of the coating application, allowing reliable protective layers to be produced using various spraying methods (Wang et al., 2021). In detonation spraying, the high particle velocity contributes to the formation of a lamellar structure with alternating areas of iron matrix and boride phases, which creates a combination of high hardness and resistance to abrasive wear with a moderate tendency to brittle fracture. This is precisely the structural organisation described in recent studies on Fe–TiB_2–CrB_2 detonation coatings, which emphasise the pronounced lamellar structure and the presence of alternating 'layers' of Fe matrix and refractory borides (Sharma et al., 2023; Liang et al., 2022).

The key problem with high-energy spraying is not the potential of the system itself, but the presence of structural heterogeneity and defects that can limit its service life: residual porosity (Rakhadilov et al., 2024), interlamellar boundaries with reduced strength, local microcracks, and 'imperfect' connectivity between the matrix and strengthening particles. For this reason, the most promising direction for Fe–CrB_2–TiB_2 coatings has become the combined route of 'detonation spraying + subsequent pulsed plasma treatment,' since post-treatment allows the surface layer to be purposefully brought to a more stable state (Tyurin et al., 2001; Pogrebnyak & Tyurin, 2003; Rakhadilov et al., 2024).

The aim of this work is to conduct a comprehensive study of the structural and phase state of detonation coatings based on the eutectic system Fe–CrB_2–TiB_2 after exposure to pulsed plasma treatment.

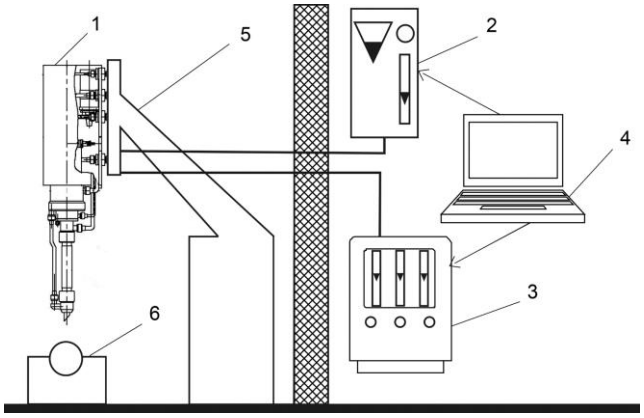
RESEARCH MATERIALS AND METHODS

Structural carbon steel 3 was used as a substrate due to its widespread use in mechanical engineering and agricultural machinery, as well as its satisfactory combination of strength characteristics, manufacturability and low cost. Before spraying, the samples were subjected to mechanical processing, degreasing and abrasive jet cleaning to ensure the required surface roughness. The functional protective layers were formed using a dispersed eutectic alloy, which is an iron-based system reinforced with refractory titanium and chromium diborides. Detailed information on the elemental composition of the cladding material is given in Table 2. The powder used consisted mainly of spherical granules, the size of which was within the range of up to 60 μm.

Table 1. Composition of Fe-TiB_2-CrB_2 powder. (wt.%):

Fe	Ni, %	Cr, %	Ti, %	B, %	Al, %
base	6÷8	20,0÷20,5	2,4÷2,5	2,5÷2,6	5,6
Note – compiled by the authors					

The coating was applied using a multi-chamber detonation device (MCDD), in which powder particles are subjected to thermal exposure and accelerated to high speeds by the energy of the detonation combustion products of a propane-butane gas mixture with oxygen and air. The parameters of detonation spraying are shown in Table 2.



1 — multi-chamber detonation atomiser; 2 — powder feeder; 3 — low-pressure gas supply panel (max. 0.3 MPa) for supplying oxygen, propane-butane and air; 4 — automated process control system; 5 — automated manipulator for moving the multi-chamber detonation atomiser; 6 — sample holder

Figure 1. Diagram of a detonation device

Note – compiled by the authors

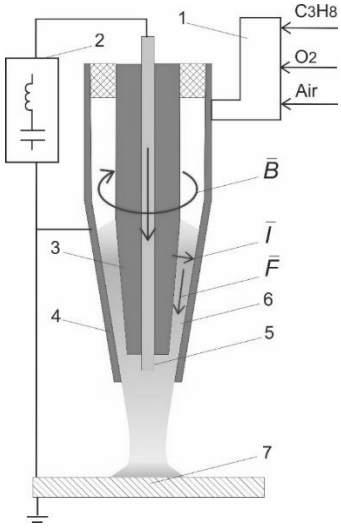
Table 2. Parameters of detonation spraying

Components of the mixture:		Consumption, m3/hour
1 chamber	O_2	2.92
	air	1.33
	C_3H_8	0.66
2 chamber	O_2	2.93
	air	1.43
	C_3H_8	0.66
Transport gas:		0,9
Note – compiled by the authors		

High-energy pulsed plasma was applied to improve the structural transformation of the formed detonation coatings. The generation of active pulses in the discharge device was based on the realization of transient detonation combustion regimes. The energy of a single pulse is approximately 4.9 kJ. The quantitative parameters of this pulsed plasma treatment (PPT) are show in Table 3.

Table 3. Parameters of pulsed plasma treatment

Parameter	Value
Voltage across the capacitor bank	3200 (V)
Capacitance of the discharge circuit capacitor bank	960 (μF)
Inductance of the discharge circuit	30(μH)
Frequency of plasma pulses	1.2(Hz)
Speed of plasma torch movement relative to the product	3,4 (mm/s)
Distance to the surface	60 (mm)
Number of passes	1
Note – compiled by the authors	



1 – detonation chamber, 2 – power source, 3,4 – coaxial electrodes, 5 – eroded electrode, 6 – plasma, 7 – sample

Figure 2. Diagram of a detonation device

Note – compiled by the authors

A X'Pert PRO system (PANalytical, Netherlands), equipped with a copper X-ray source, was used to perform the phase analysis. The experiment was carried out at operating parameters of 40 kV and 30 mA. Diffraction data in the Cu−Kα spectrum (λ=1.541 Å) were collected over the range 2θ=20–90°. The measurement was carried out using a step-by-step scanning method with a step size of 0.02°, with a signal exposure time of 0.5 s at each point. The experimental diffractograms were interpreted and phase identification was performed using the HighScore Plus software package (version 3.0.5). Scanning electron microscopy, carried out using a Tescan Vega 4 system (Czech Republic), was employed to study the morphology of the cross-sections of the formed protective layers. The anti-corrosion properties were evaluated in an electrolytic cell using a CS300 potentiostat-galvanostat. An aqueous sodium chloride solution (3.5% NaCl) was used as the working medium at a stabilised temperature of 25±1 °C. Measurements were carried out using the classical three-electrode configuration, where a silver-chloride cell (Ag/AgCl) served as the reference electrode and a platinum plate as the auxiliary electrode. Quantitative indicators of corrosion resistance, including the corrosion potential (E_(corr)) and current density (i_(corr)), were calculated based on the analysis of potentiodynamic curves using the Tafel extrapolation method. Polarisation curves were recorded over a potential range of ±0.1 V relative to the open-circuit potential (OCP) at a constant scan rate of 0.5 mV/s. To increase the statistical reliability of the study, it was performed on four samples with three repetitions for each. The experimental results were processed using the specialised software environment CS Studio 6, whose algorithms enable the automatic identification of the working segments of the Tafel curves and the calculation of key electrochemical constants. The microhardness values of the formed coatings were measured using the FISCHERSCOPE HM2000S measuring system (Helmut Fischer GmbH, Germany) in accordance with ASTM E2546. To achieve high reliability and account for possible structural heterogeneity in different zones of the cross-section, at least ten indentation tests were performed for each sample, followed by mathematical averaging of the final data. The tests were carried out at a load of 100 mN and a holding time of 10 s. The amount of wear was determined using a Leica DCM8 non-contact optical profilometer (Leica Microsystems, Wetzlar, Germany). Roughness parameters were evaluated using confocal microscopy with a Leica EPI 20× lens and Leica Scan 6.5 software. The obtained profilometric data were processed and analysed using the Mountains software package.

RESULTS AND DISCUSSION

Figure 3 shows the X-ray diffraction spectra of coatings formed from an eutectic Fe–TiB_2–CrB_2 alloy using detonation spraying. The coating in its initial state (Fig. 3a) is characterised by the dominance of intense α-Fe lines, which indicates the predominant role of the ferrite base in the material structure. In addition, reflections of γ-Fe and the $Cr_{23}C_6$ carbide phase formed during the spraying process are observed. At the same time, the peaks corresponding to the refractory boride phases TiB_2 and CrB_2 are weakly expressed, which indicates the limited development of the eutectic structure due to the ultra-rapid cooling of the molten particles. After pulsed plasma treatment (Fig. 3b), a significant change in the phase composition is observed. The intensity of the TiB_2 and CrB_2 diffraction maxima increases noticeably, while the content of γ-Fe and $Cr_{23}C_6$ decreases. This transformation is due to the effect of high-energy pulses, which promote the additional formation of boride phases and structural relaxation of the coating. As a result of the modification, a more uniform eutectic structure develops, characterised by an increased concentration of dispersed high-hardness TiB_2 and CrB_2 particles distributed in the iron matrix.

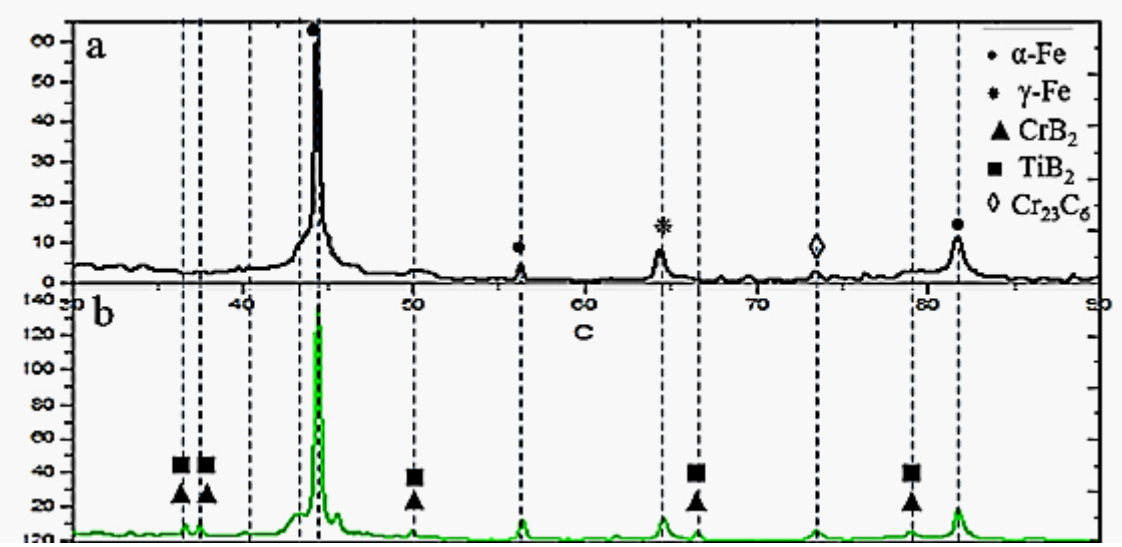


Figure 3. Results of X-ray diffraction analysis of Fe-TiB2-CrB2 coatings before (a) and after pulse plasma treatment (b)

Note – compiled by the authors

Figure 4 shows three-dimensional topographical images of wear traces on the Fe–TiB_2–CrB_2 coating system in its initial state (Fig. 4a) and after pulsed plasma treatment (Fig. 4b). The coating in its initial state (Fig. 4a) is characterised by the formation of a pronounced wear track with a noticeable depth and uneven profile. The 3D map shows local depressions and pronounced ridges at the edges of the track, indicating the development of plastic deformation and microcutting. The profilogram shows a significant amplitude of height fluctuations, indicating increased volumetric wear and unstable destruction of the surface layer. After pulse plasma treatment (Fig. 4b), the shape of the wear track becomes more uniform. The depth of the track decreases, the profile smoothes out, and the height differences at the edges of the track are reduced. The 3D image shows a more uniform surface without pronounced areas of chipping. This indicates a reduction in wear and an increase in resistance to plastic deformation. The results of tribological tests show that in the initial sample (before treatment), the average coefficient of friction of the coatings is ~0.56, whereas after PPT treatment, the coefficient of friction decreases to 0.53

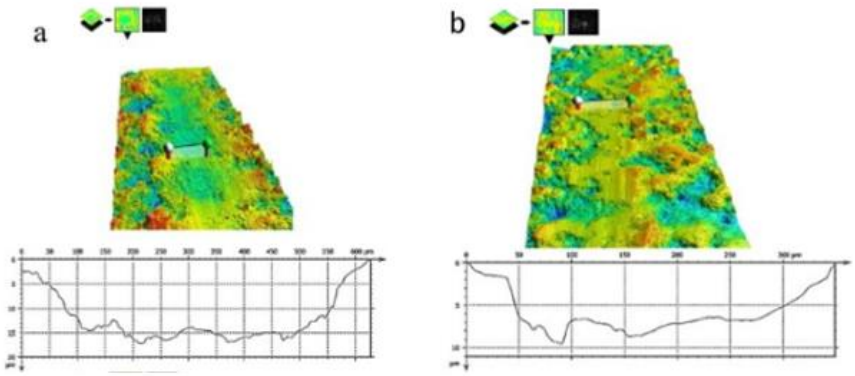


Figure 4. Three-dimensional topographical images of wear traces on coatings in their as-received state (Fig. 4a) and after pulsed plasma treatment (Fig. 4b)

Note – compiled by the authors

Figure 5 shows the potentiodynamic polarisation curves of the Fe–TiB_2–CrB_2 coating obtained under standard three-electrode electrochemical cell conditions. After pulse plasma treatment (curve b), a noticeable shift of the corrosion potential to a more positive region is observed. The observed effect confirms an increase in the coating's corrosion resistance and an improvement in the condition of the surface layer. The decrease in anode current density indicates a decrease in the rate of electrochemical dissolution of the metal matrix due to structural transformations. The increase in corrosion resistance after pulsed plasma treatment is associated with a number of factors. Firstly, the surface layer is compacted and the open porosity is reduced, which limits the penetration of the electrolyte into the deeper areas of the coating. Secondly, grain refinement and stabilisation are observed, ensuring a more uniform distribution of potential across the surface. Thirdly, the microgalvanic interaction between α-Fe and the dispersed boride phases TiB_2 and CrB_2 is reduced. Together, this leads to the formation of a denser and chemically stable surface with a reduced rate of electrochemical degradation. A shift in corrosion potential of 0.15–0.20 V confirms the effectiveness of structural optimisation of the coating without significantly changing its phase composition. The electrochemical test data are consistent with the results of X-ray phase analysis, which demonstrate the preservation of the TiB_2 and CrB_2 phases with a simultaneous reduction in the γ-Fe content.

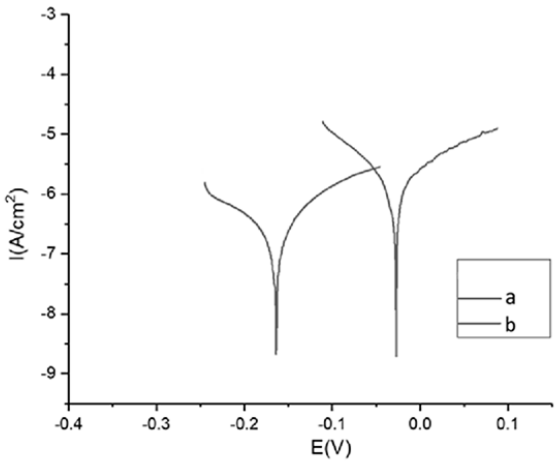


Figure 5. Results of analysis with CS300 potentiostat-galvanostat coatings before (a) and after pulse plasma treatment (b).

Note – compiled by the authors

Figure 6 shows the microhardness distribution profiles with respect to coating depth. The initial state is characterised by a maximum hardness value in the near-surface region, reaching approximately 15 GPa. As the indenter penetration depth increases, a gradual decrease in hardness to a level of 13–12 GPa is observed, which is associated with the transition from the modified surface layer to the bulk of the coating. This dynamic is due to the structural features of the detonation coating, including its lamellar structure, the presence of micropores, and the limited degree of interparticle sintering. The application of pulsed plasma modification results in a significant increase in microhardness in the surface layer to 17–18 GPa, which exceeds the initial values by 15–20%. At depths exceeding 100–150 μm, the values stabilise at 12–13 GPa, indicating that the hardening is local in nature, confined exclusively to the heat-affected zone of the plasma discharge. The structural causes of the improvement in mechanical properties are the intensive compaction of the material, grain refinement, and the uniform distribution of dispersed boride and carbide phases, which is fully confirmed by the results of X-ray structural analysis. Furthermore, an increase in the cohesive strength of the coating is observed due to the elimination of surface defects and the 'healing' of interlamellar boundaries upon their partial melting. The sharp drop in hardness at the end of the measurement profile (deeper than 180 μm) is due to the fact that the deformation zone beneath the indenter reaches the metal substrate, which has significantly lower stiffness compared to the protective layer.

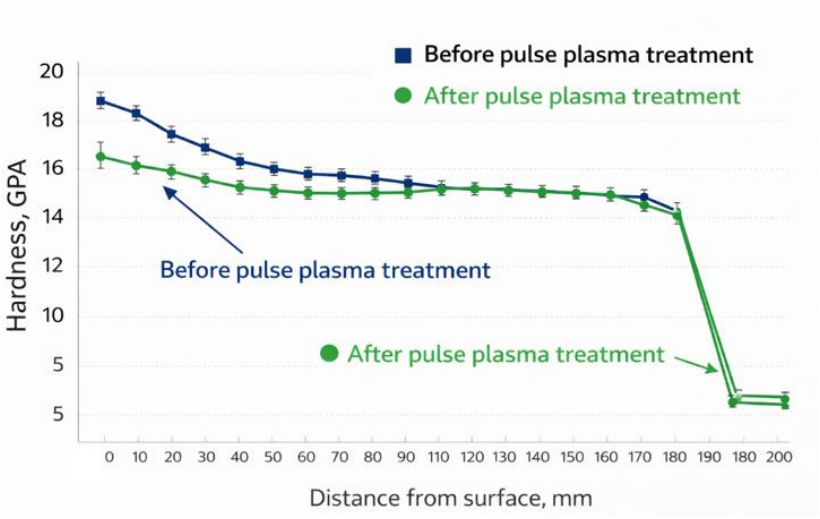


Figure 6. Hardness test results

Note – compiled by the authors

Figure 7 illustrates changes in the morphology of cross-sections of the iron-boride eutectic-based coating under the influence of PPT (7b) compared with the initial state (7a), as well as data from linear scanning of the chemical composition. In the post-deposition state (7a), the layer exhibits a lamellar architecture typical of gas-detonation spraying technologies. The boundaries of the sprayed particles, interlamellar layers and local micropores are clearly visible. The structure is heterogeneous, with some areas having different densities. According to the data of linear elemental analysis, a relatively uneven distribution of alloying elements is recorded, which is due to the ultra-fast crystallisation of molten particles during deposition. A sharp transition in chemical composition is observed near the 'coating-substrate' boundary.After pulsed plasma treatment (Fig. b), the morphology of the cross-section changes significantly. The structure becomes denser and more homogeneous, the interlamellar boundaries are partially smoothed, and the number of visible micropores decreases. The surface layer acquires a more compact

structure due to local remelting and subsequent recrystallisation. The linear distribution of elements demonstrates more stable concentration profiles without pronounced fluctuations, which shows a more uniform distribution of chemical elements within the heat-affected zone.

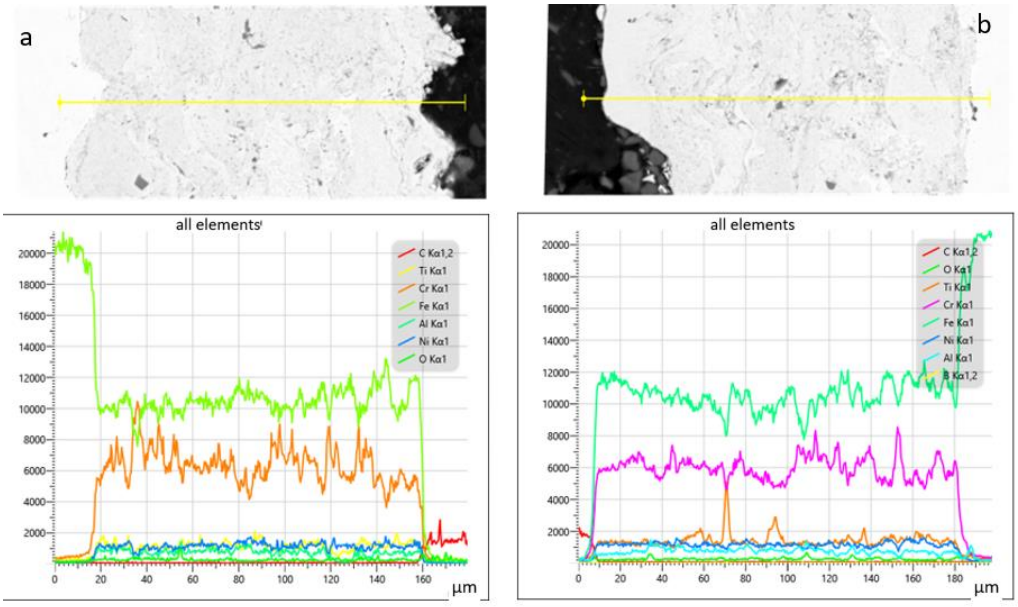


Figure 7. Cross-sectional morphology of coatings before (a) and after pulse plasma treatment (b).

Note – compiled by the authors

CONCLUSION

Based on the results of X-ray structural analysis, microstructural studies, microhardness measurements, tribological and corrosion tests, the following was established.

- X-ray phase analysis showed that after PPT, the phase composition of the coating (α-Fe, TiB_2, CrB_2) is preserved, while phase redistribution and a decrease in the proportion of γ-Fe and the $Cr_{23}C_6$ carbide phase are observed. At the same time, the intensity of boride phase reflections increases, indicating structural stabilisation and the development of eutectic morphology without phase degradation.

- SEM analysis of the cross-section confirmed that pulse plasma treatment leads to compaction structural densification of the coating, reduction in interlamellar porosity, and local melting of surface defects. A more homogeneous and compact surface layer with reduced concentration heterogeneity is formed.

- Microhardness profiles showed a significant increase in hardness in the near-surface zone — from ~15 GPa to 17–18 GPa (an increase of 15–20%). The hardening is localised and limited to the zone of thermal exposure of the plasma pulse. The increase in hardness is associated with the compaction of the structure, grain refinement and an increase in the proportion of dispersed boride phases.

- The results of tribological tests indicate a reduction in the depth and volume of wear after treatment, which indicates an increase in the wear resistance of the coating. The wear mark becomes more uniform, and the intensity of microcutting and plastic deformation decreases.

- Corrosion studies have shown a shift in the corrosion potential to the positive region by 0.15–0.20 V and a decrease in the rate of anodic dissolution. The improvement in corrosion resistance is due to a decrease in open porosity, a decrease in microgalvanic activity between phases, and the formation of a denser and chemically stable surface.

Thus, pulsed plasma treatment is an effective method for modifying Fe–TiB_2–CrB_2 detonation coatings, providing structural optimisation without changing the phase composition and leading to a simultaneous increase in hardness, wear resistance and corrosion resistance..

CONFLICT OF INTEREST: The authors declare that there is no conflict of interest..

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