



## TRIBOLOGICAL WEAR OF FE-AL COATINGS APPLIED BY GAS DETONATION SPRAYING

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### Abstract

Comparative tests of gas detonation (GDS) coatings were carried out in order to investigate the influence of spraying parameters on abrasive wear under dry friction conditions. The tests were carried out using the pin-on-disc (PoD) method at room temperature. The microstructure of the coatings was analysed by X-ray diffraction (XRD) and scanning electron microscopy (SEM/EDS) methods. The results showed that with certain parameters of the GDS process, the main phase of the produced coatings is the FeAl phase with the participation of thin oxide layers, mainly  $Al_2O_3$ . The tribological tests proved that the coatings sprayed with the shorter barrel of the GDS gun showed higher wear resistance. The coefficient of friction was slightly lower in the case of coatings sprayed with the longer barrel of the GDS gun. During dry friction, oxide layers form on the surface, which act as a solid lubricant. The load applied to the samples during the tests causes shear stresses, thus increasing the wear of the coatings. During friction, the surface of the coatings is subjected to alternating tensile and compressive stresses, which lead to delamination and is the main wear mechanism of the coatings.

## Introduction

Despite the low production costs, the industrial application of solid FeAl alloys is limited due to low ductility and resistance to cracking at room temperature. The research proved that Fe-Al coatings sprayed with supersonic methods solve the problems encountered in the production of these alloys using the traditional method (CHROSTEK 2020, SENDEROWSKI et al. 2016). In addition, the Fe-Al phase-matrix intermetals produced by the GDS method are a material with unique properties. They are resistant to high-temperature corrosion (heat resistance) in aggressive sulphide and chloride environments (SENDEROWSKI 2015). This creates potential opportunities for their use as heat-resistant construction materials (PANAS et al. 2019).

The reason for this is that the FeAl powder particles are subjected to strong oxidation in a hot stream of gaseous products of supersonic combustion detonation. This results in the formation of a multiphase coating structure with the participation of oxide phases formed at the grain boundaries in the form of thin films, due to the strong plastic deformation of the powder particles forming the coating. The grains of primary particles change their morphology from equiaxial to streaked during strong plastic deformation (CHROSTEK 2020, FIKUS et al. 2019, SENDEROWSKI et al. 2011).

Most of the research work focuses on the characteristics of the microstructure and thermophysical properties of the resulting coatings, forgetting about their functional properties. (BINSHI et al. 2004). However, from such coatings, above all, high wear resistance is expected (BOJAR et al. 2002), therefore the aim of this article is to investigate and compare the dry friction abrasive wear of GDS spray coatings with different spraying parameters.

## Materials and Method

The research was carried out on intermetallic protective coatings produced by the GDS method from alloy powder on a FeAl phase matrix with the composition Fe<sub>40</sub>Al<sub>10.05</sub>Zr % at. and 50 ppm B, produced by the company LERMPS-UTBM by the VIGA method (Vacuum Induction Melting and Inter Gas Atomization). The base material is 13CrMo4-5 (15HM) boiler steel with dimensions of 50×50×5 mm, which was blasted with electro corundum immediately before the spraying process. The surface roughness after sandblasting of the substrate was  $Ra = 18.98 \mu\text{m}$ . The coating in the form of a circular deposit (CHROSTEK 2020) was sprayed with the substrate stationary in relation to the barrel of the detonation gun operating at a frequency of 6.66 Hz. The barrel of the GDS gun was positioned at a distance  $L = 110 \text{ mm}$  of from the sprayed surface. Two barrel lengths were used, 590 and 1090 mm. All the GDS spraying parameters presented

above, together with the composition of the explosive detonation mixture and the flow of air transporting the powder, are presented in Table 1. The GDS spraying of the FeAl coating was performed by the Department of Protective Coatings – E.O. Paton Electric Welding Institute of the National Academy of Sciences of Ukraine, using the “Perun S” detonation gun.

Table 1

GDS spraying parameters				
Powder Fe40Al10,05Zr at. %+50 ppm B; particle size distribution (granulation) 5-40 $\mu\text{m}$				
Powder transporting gas – air			0.4 $\text{m}^3/\text{h}$	
Oxygen-fuel mixture			C <sub>3</sub> H <sub>8</sub> – 0.45 $\text{m}^3/\text{h}$ O <sub>2</sub> – 1.52 $\text{m}^3/\text{h}$ air (as diluter gas) – 0.65 $\text{m}^3/\text{h}$	
Spraying frequency			$f = 6.66 \text{ Hz}$	
Coating	spraying distance $L$ [mm]	barrel length [mm]	PIP* [mm]	number of GDS shots
A	110	590	274.5	100
B		1,090	274.5	400
C		1,090	412.5	100
D		590	412.5	400

\* powder injection position – place of the introduction of the powder into the barrel at the time of detonation

The structural tests of the coatings were carried out using scanning electron microscopy with X-ray microanalysis (SEM/EDS) and X-ray diffraction (XRD). The point analysis and surface distributions of specific alloying elements were performed on a Quanta 3D FEG Dual Beam high-resolution scanning electron microscope with SE (secondary electron detector) and BSE (backscattered electron detector) detectors. SEM/EDS chemical composition studies in micro-areas were carried out using the EDAX Genesis Spectrum analyzer.

XRD tests were carried out using a Rigaku Ultima IV diffractometer with a  $\text{CoK}_\alpha$  monochromatic radiation focusing beam with a spectral wavelength  $\lambda = 0.178897 \text{ nm}$ . Filtering corresponding to the  $\text{CoK}_\alpha$  wave was used with the lamp operating conditions of 40 kV/40 mA. A record was made in the angular range from  $20^\circ$  to  $120^\circ$  with a scanning speed of  $1^\circ/\text{min}$ .

Microhardness measurements were made using the Vickers method using the Innovatest 400-DAT microhardness tester, at a load of 0.98 N (HV0.1) for 10 s, in accordance with the PN EN ISO 6507-1: 2007 standard. The research was carried out in a cross-section on polished metallographic specimens in a plane perpendicular to the applied Fe-Al coatings. The microhardness distribution

measurements were carried out with a step of 0.1 mm from the substrate towards the coating surface along three measurement paths spaced 0.1 mm apart.

Abrasive wear tests in dry friction conditions using the pin-on-disc method were performed on a Tribotester T-10 in dry sliding conditions at room temperature (20°C). The relative humidity of the ambient atmosphere was 50%. The sample was a pin with a diameter of  $\varnothing 7$  mm. Two samples were taken from each coating, which constituted a significant area of it (the diameter of the coating was  $\varnothing 25$  mm). EDM cutting was used. A pearlitic cast iron disc with a hardness of 33 HRC was used as a counter-sample. The radius of rotation was  $r = 18$  mm, which at the rotational speed of  $v_r = 48$  rpm gave the sliding speed  $v = 0.09$  m/s. The total friction path was 1,040 m, which each sample traveled during  $t = 11,500$  s (approx. 3 h). The pins were subjected to a load of  $F = 20$  N. The coefficient of friction was computer-monitored during the test by measuring the elastic deflection of the arm. The T-10 device is equipped with a measurement and control system, which includes: a set of measuring transducers, a computer with dedicated measurement and recording software (Fig. 1a, b). During the course of the test, the wear products from the friction junction were not removed in order to best reflect the actual conditions. Wear products always remain between the two materials during friction (Fig. 1c).

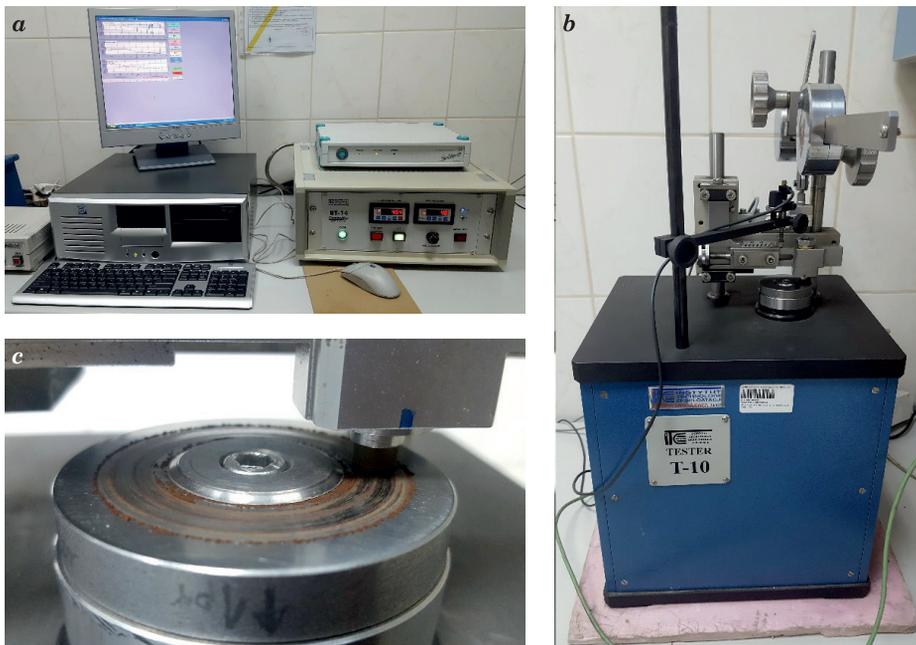


Fig. 1. Apparatus for abrasive wear tests using the pin-on-disc method: a – computer with a control controller, b – T-10 tester, c – visible oxidation of the surface layer of metal elements during the pin-on-disc measurement

## Results and Discussion

The XRD tests of the coatings (Fig. 2) indicate the presence of the FeAl phase as the basic component of the structure. At the same time, the share of Fe<sub>3</sub>Al phase was confirmed. The presence of oxide phases FeO, Fe<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub> and spinel Fe(Al<sub>2</sub>O<sub>4</sub>) was also detected.

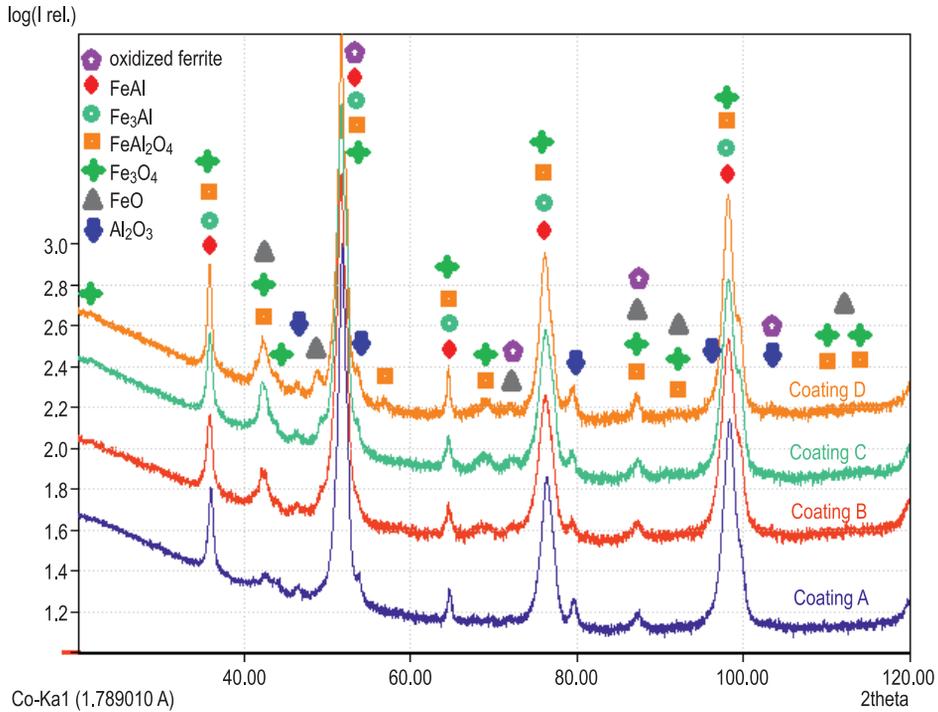


Fig. 2. XRD analysis of the phase composition of FeAl coatings produced by the GDS method

Figure 3a shows a photo of the SEM/EDS microanalysis that was performed on the surface of the coating. The powder particles melt or completely melt, resulting in a strong oxidation of the diffusing aluminum and the formation of FeO (1) and Al<sub>2</sub>O<sub>3</sub> (2) oxide phases on the surface of the molten particles. Structural studies carried out on the cross-section (Fig. 3b, c) show a typical lamellar structure with different (multi-phase) chemical composition, where we can distinguish the basic phase FeAl (3), Al<sub>2</sub>O<sub>3</sub> (6), oxidized ferrite (1), Fe<sub>3</sub>Al (2), FeO, Fe<sub>3</sub>O<sub>4</sub> (4) and Fe(Al<sub>2</sub>O<sub>4</sub>).

The content of alloy elements and oxygen mapped in the SEM/EDS microanalysis of chemical composition at the cross-section of FeAl coating (GDS) is presented in Table 2.

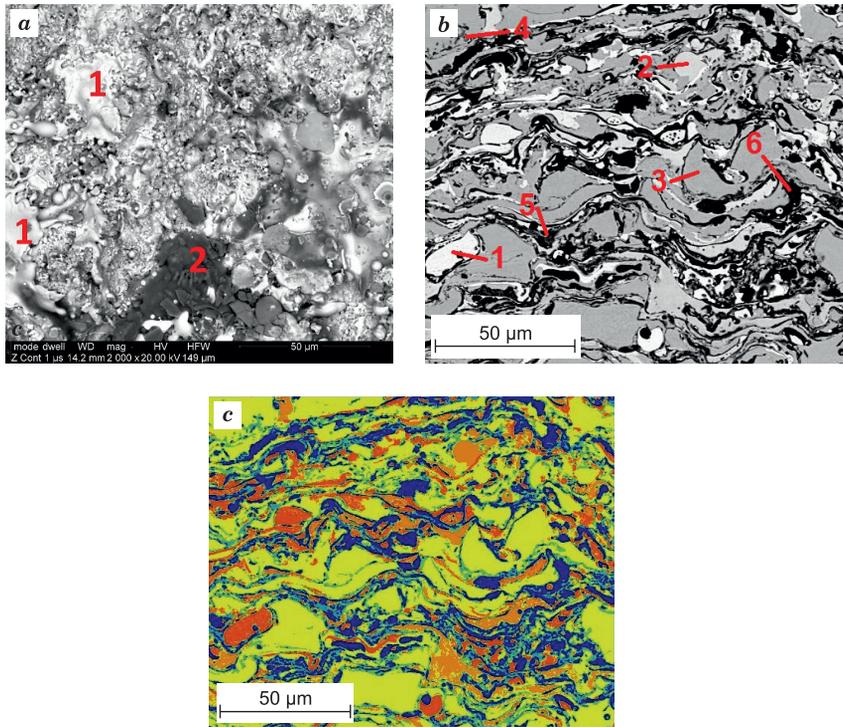


Fig. 3. SEM/EDS microanalysis of Fe-Al coatings formed in the GDS process: *a* – coating surface, *b* and *c* – coating cross-section

Table 2

Content of alloy elements and oxygen mapped in the SEM/EDS microanalysis of chemical composition at the cross-section of FeAl coating (GDS) (Fig. 3c)

Analyzed region on coating surface	Content of alloy elements [% at.]			Probable phase
	Color	Fe	Al	
Blue	0.89	48.02	51.09	Al <sub>2</sub> O <sub>3</sub> phase
Light blue	22.10	35.12	42.78	Fe(Al <sub>2</sub> O <sub>4</sub> ) phase
Green	49.21	11.16	39.63	FeO, Fe <sub>3</sub> O <sub>4</sub> oxide phases
Yellow	55.88	40.59	3.53	weakly oxidized FeAl phase
Orange	76.86	18.79	4.35	weakly oxidized Fe <sub>3</sub> Al phase
Red	92.64	1.14	6.22	oxidized ferrite

The presence of very hard oxide phases in the coating structure has a significant impact on the degree of hardening of the produced coatings. For this purpose, the cross-sectional microhardness of the coatings was tested by making three measurement paths from the steel substrate to the top layer of the coating, at intervals of 0.1 mm (Fig. 4).

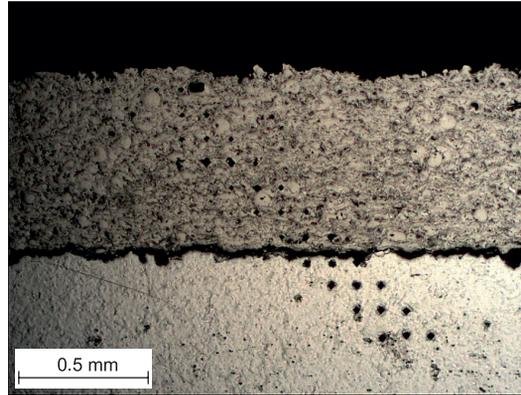


Fig. 4. Measurements of microhardness using the Vickers method on the cross-section of the coating A

The results obtained (Fig. 5) show a large difference in hardness in the multiphase coating structure from about 300 to 650 HV<sub>0,1</sub> (ignoring the extremely low values caused by the porosity of the coating). The highest values are shown in strongly oxidized (dark) areas. The areas with phases with high iron content

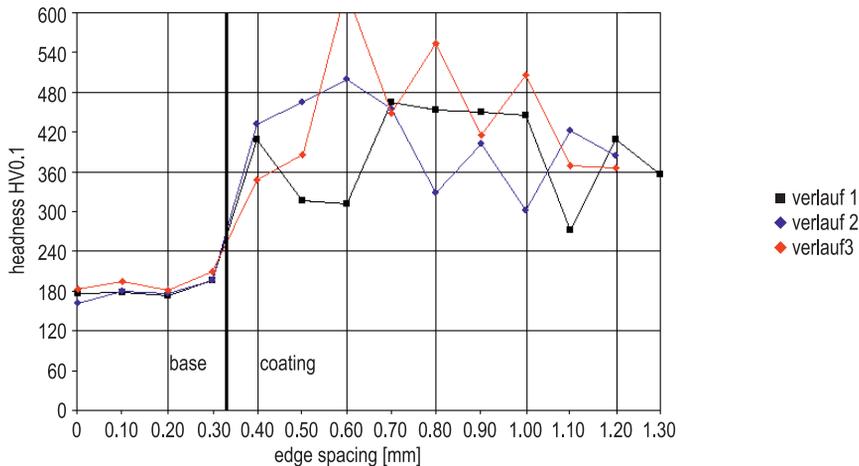


Fig. 5. Distribution of the microhardness HV<sub>0,1</sub> of coating B along three parallel measuring paths

and low oxygen content show the lowest hardness. The average value of the microhardness of coatings made with a shorter barrel (590 mm) is 448 HV0.1, while the microhardness of coatings made with a longer barrel (1,090 mm) is 427 HV0.1. These values are much higher than the microhardness of the powder charge  $230 \pm 10$  HV0.1 (Fig. 6).

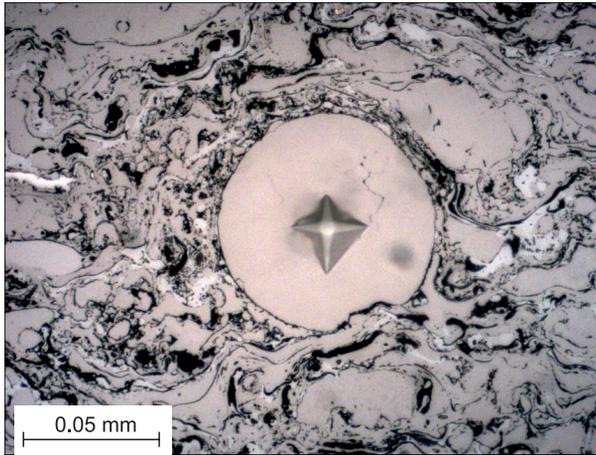


Fig. 6. An example of a microhardness measurement carried out using the Vickers method on the cross-section of an unfused powder particle, performed on coating A

The oxide phases  $\text{Al}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{Fe}(\text{Al}_2\text{O}_4)$  occurring in the coating, also dispersed in micro-areas, are the main reason for the high hardness of intermetallic coatings produced by the GDS method.

The pin-on-disc (PoD) method is a commonly used technique to determine the friction coefficient  $\mu$  and wear under various tribological conditions. These studies allowed to determine the influence of the oxide phases on the functional properties of the cermet structure of the coating under conditions of abrasive wear during dry friction.

Figure 7 shows the evolution of the friction coefficient  $\mu$  as a function of the sliding distance. All tests showed a similar change in the coefficient of friction. In the first phase, an increase is visible up to about 15 minutes, followed by a second stabilization phase with slight fluctuations in value. The first phase is a typical run-in phenomenon where the surface topography changes until the system reaches steady state.

The high hardness of GDS coatings, determined by the structure with the participation of oxide ceramics, is also the direct cause of the high abrasive wear resistance of this type of coating under dry friction conditions. It can be assumed that the coefficient of friction  $\mu$  (Fig. 8) would be higher with

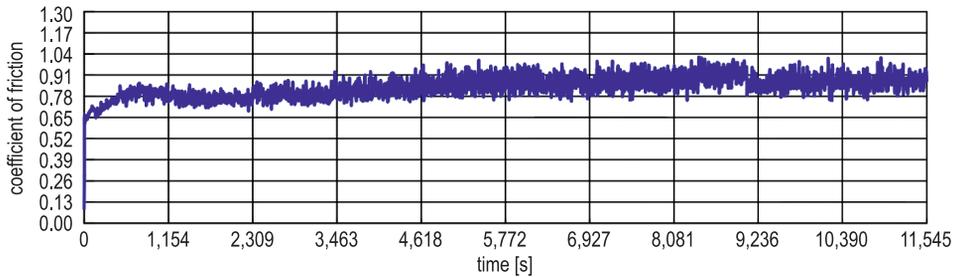


Fig. 7. Spindle sliding wear test on the disc: coefficient of friction as a function of the slip time of the coating  $D$  produced by the GDS method

the application of lower loads (the tests were carried out with a high unit load of the sample with the force  $F = 20$  N). As the load increases, the friction coefficient decreases slightly due to the increase in the friction contact temperature and the formation of larger surfaces of the oxide layer on the worn surface, which acts as a lubricant (Fig. 9) (BINSHI et al. 2004).

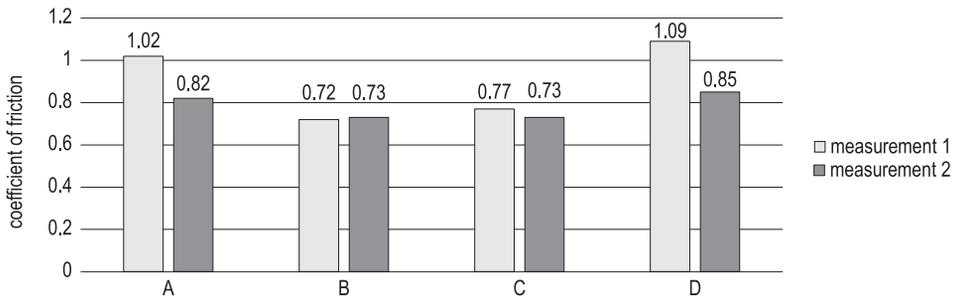


Fig. 8. Average coefficients of friction of the tested coatings at a constant load of  $F = 20$  N

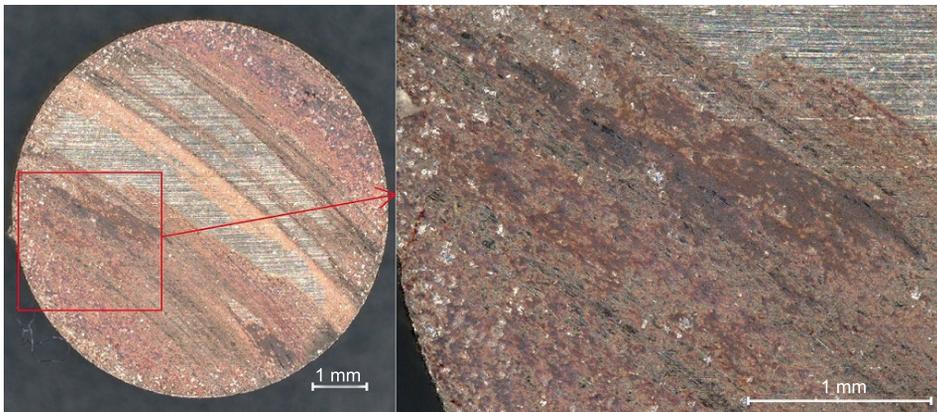


Fig. 9. Fe-Al coating of sample  $B$  after tribological tests with a visible oxide film on the surface

The sliding wear test shows changes in the wear rate of Fe-Al coatings under constant load. As shown in Figure 10, the greatest increase in wear occurs in the first stage of the test (up to about 10 minutes). Then the formation of a layer of oxides detaching from the surface of the sample slows down this process. The excess of accumulated powdered material (visible in the graph in the range from 2,309 s to 3,463 s) is pushed to the sides (Fig. 1c), which contributes to a further increase in consumption.

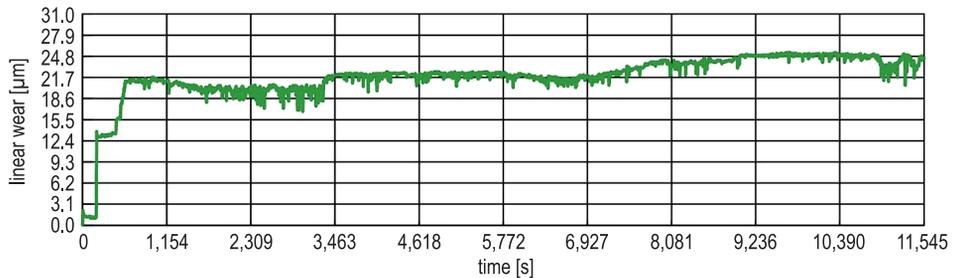


Fig. 10. Pin sliding wear test on the disc: linear wear as a function of the slip time of the coating C produced by the GDS method

The results of the research revealed that the length of the detonation gun barrel was of considerable importance for the strengthening of the structure. The coefficient of friction is clearly higher in the coatings sprayed with a shorter barrel (590 mm). This is also reflected in the wear of the coatings. The consumption of coatings A and D is much lower (Fig. 11). This shows that the use of a shorter barrel clearly increases the wear resistance of the GDS coating, despite the similar proportion of oxide phases in all tested coatings.

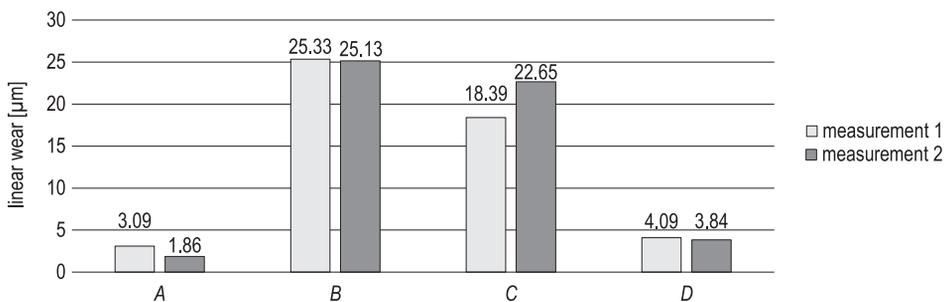


Fig. 11. Average rates of linear wear of the tested coatings at a constant load of  $F = 20$  N

Despite the low porosity (SENDEROWSKI 2015), individual grains are torn out of the matrix (Fig. 12). The reasons for this are: the high coefficient of friction ( $\mu = 0.72-1.09$ ) (Fig. 8) and the brittleness of this type of coatings due to the high percentage of oxide ceramics. In addition, high loading causes maximum

shear stress, thereby increasing wear. During sliding, the surface of the coating is subjected to alternating tensile and compressive stresses, so delamination seems to be the dominant wear mechanism of the coatings.

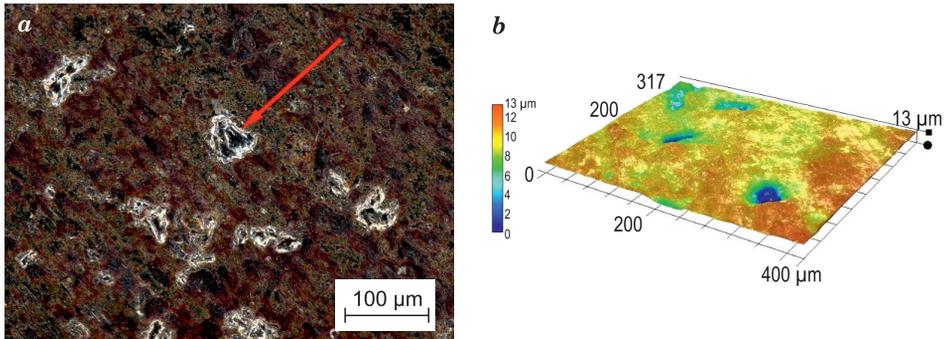


Fig. 12. Morphology of Fe-Al coatings of sample *B* produced by GDS method after wear tests: *a* – visible grains torn out from the matrix, *b* – 3D map of the area under study

## Conclusions

Tribological tests carried out on coatings produced with the gas detonation method (GDS) under dry friction conditions made it possible to compare the wear of the coatings with the use of various spraying parameters.

All tested coatings, regardless of the spraying parameters, have a lamellar structure, typical for coatings sprayed with supersonic methods. The basic structure is the FeAl phase. During the formation of the coating, changes occur with the participation of oxygen, during which oxide phases  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ , FeO,  $\text{Fe}(\text{Al}_2\text{O}_4)$  and phases poor in iron or aluminum are formed.

Coatings sprayed with a shorter barrel, 590 mm long, showed significantly higher wear resistance, despite the fact that the coefficient of friction was similar for both groups of materials.

Compressive and tensile stresses acting on the samples during the tests, as well as oxidized wear products in the friction area, led to material chipping, which is the main wear mechanism of the coatings. No cracks were observed in the coating structure.

The research proved that with properly selected spraying parameters, Fe-Al alloys in the form of protective coatings can have high abrasion resistance, also at room temperature.

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