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Experimental investigations of Al₂O₃- and ZrO₂-based coatings deposited by detonation sprayingBauyrzhan Rakhadilov¹, Nurgamit Kantay², Zhuldyz Sagdoldina¹, Dosym Erbolatuly¹, Gulsym Bektasova¹ and Maciej Paszkowski³ ¹ S. Amanzholov East Kazakhstan University, 070002, Ust-Kamenogorsk, 18/1 Amurskaya St., Kazakhstan² D. Serikbayev East Kazakhstan Technical University, 070003, Ust-Kamenogorsk, 19 Serikbayev St., Kazakhstan³ Wrocław University of Science and Technology, Wybrzeże Stanisława Wyspiańskiego 27, 50-370 Wrocław, PolandE-mail: maciej.paszkowski@pwr.edu.pl**Keywords:** coatings, detonation spraying, microstructure, hardness, tribology

Abstract

This paper presents the results of experimental investigations of the physical and mechanical properties, structure and surface morphology of Al₂O₃- and ZrO₂-based coatings deposited by detonation spraying. The coatings were sprayed onto titanium-alloyed steel 12Ch18N10T (GOST 4986-79). The thickness of the coatings ranged from 250 to 1100 μm . After the deposition of the coatings the mechanical properties of the steel's surface layer significantly improved. Its microhardness increased threefold, reaching the maximum value of over 11 GPa. Also the tribological properties of the material detonation sprayed onto the steel were tested. A significant decrease in the kinetic friction coefficient was observed. For the steel with the ZrO₂ coating the friction coefficient decreased by nearly half, reaching the value of 0.330. An increase in abrasive wear resistance was noted. Moreover, the results of electron microscopy, energy-dispersive X-ray spectroscopy and X-ray diffraction examinations evaluating the structural properties and surface morphology of the coatings are presented.

1. Introduction

Engineering materials are often exposed to damage caused by mechanical loads, thermal radiation and/or an aggressive environment. Bearing this in mind, not only novel materials, but also technologies significantly improving the performance of the currently used materials are developed. By covering the work surfaces of machine parts with coatings with special performance characteristics one can significantly extend the service life of the machines. In such a case, a part can be made of a lower-quality and cheaper material and only the part's crucial places will be improved by applying a suitable coating. One of the more dynamically developing coating deposition technology is thermal spraying. Using this technology one can produce high quality durable coatings. Among thermal spraying methods, detonation spraying (DS) and high velocity oxygen fuel (HVOF) spraying are the most effective techniques of producing hard, dense and wear-resistant coatings [1–6]. DS and HVOF are characterized by a high velocity and a relatively low temperature, whereby the feedstock powder is only minimally oxidized and decomposed in the process [7, 8]. Coatings are widely applied to automotive parts, boiler components, power equipment, chemical process equipment, medical equipment, aircraft engines, land and offshore turbines, in the shipbuilding industry, etc [9–11]. Al₂O₃ and ZrO₂ oxides are often used in the manufacture of heat-resistant ceramic coatings. The two oxides show similarly good properties at high temperatures. Al₂O₃ is more available than other oxides. Thermally sprayed Al₂O₃ consists of different phases, i.e. α , γ , δ , θ , etc, among which the α and γ phases predominate. Which of the phases form depends on the nature of the feedstock material and its characteristics during melting and solidification [12]. Using the detonation spraying technique one can obtain a coating with excellent adhesive strength, low porosity and low residual compressive stresses [13, 14]. Under the influence of detonation waves the particles of the sprayed powder reach the substrate at a speed of 800–1200 m s^{−1} [15, 16]. The quality of detonation sprayed coatings



Figure 1. Detonation spraying unit CCDS-2000.

very much depends on the surface roughness of the coating material (the degree of its processing) and its chemical composition, the size of the granules, the ratio of the gases, and the impurities. In many cases, the feedstock powder consists of only α - Al_2O_3 . Then the presence of α - Al_2O_3 after detonation spraying depends on the presence of insoluble or semi-molten particles in the powder. γ - Al_2O_3 forms as a result of the rapid crystallization of the molten volume [17]. It has been shown that the addition of such powders as TiO_2 , Cr_2O_3 , SiC , ZrO_2 , etc improves the properties (stability) of α - Al_2O_3 [18–20]. As part of the present study the structural properties and surface morphology of Al_2O_3 and ZrO_2 ceramic coatings deposited by detonation spraying were investigated. The coatings' basic mechanical properties, including microhardness, and their tribological properties and abrasive wear resistance were evaluated.

2. Test material

Detonation spraying was employed as the method of producing Al_2O_3 and ZrO_2 coatings. Alumina powder (20–60 μm) and zirconium powder (20–80 μm) were used. Spraying was carried out using a CCDS2000 detonation gun with an 800 mm long 20 mm diameter barrel (figure 1), installed in the Research Centre for Surface Engineering and Tribology, Kazakhstan.

Samples made of titanium-alloyed austenitic stainless steel 12Ch18N10T, corresponding to steel AISI 321, were used as the substrate for coating deposition. The chemical composition of the tested steel is specified in standard GOST 4986-79 [21]. The steel samples' dimensions were 75 × 50 × 5 mm and their roughness was $R_a = 0.088 \mu\text{m}$. First the substrates were chemically cleaned for 7–10 min. Then the dried substrates were sandblasted (using Contracor Eco140S, Germany) with electrocorundum with a grain size of about 300 μm to achieve an average roughness of 4.5 μm . The spraying parameters and the surface roughness after detonation spraying are shown in table 1.

3. Test method

The surface morphology, the fracture fractograms and the friction path were examined under a Phenom ProX scanning electron microscope (Phenom-World BV, Netherlands). The elemental composition of the coatings was analysed on the basis of their characteristic X-ray spectra by means of an energy-dispersive X-ray spectrometer (EDS) built into the microscope. X-ray diffraction analyses of the coated samples were carried using an Xpert PRO diffractometer ($U = 40 \text{ kv}$, $I = 30 \text{ mA CuK}\alpha$) in the dot-wise mode with a scanning step of $2\theta = 0.02 \text{ deg}$. (PANalytical, Netherlands). Also the mechanical properties of the coatings were tested. Hardness—a measure of a material's resistance to local plastic strains—is the basic parameter characterizing coatings. The microhardness of the coatings was measured under an indenter load of 0.05 N by means of a DuraScan 20 G5

Table 1. Detonation spraying parameters and surface roughness of detonation sprayed Al_2O_3 and ZrO_2 coatings.

Coating	Barrel filling ($\text{C}_2\text{H}_2/\text{C}_3\text{H}_8/\text{N}_2/\text{O}_2$), %	Powder dispenser filling, %	Thickness, μm	R_a , μm
Al_2O_3	71	66	250	5.10
			500	2.67
			1100	2.20
ZrO_2	81	41	250	1.79
			500	2.29
			1000	4.62

automated hardness tester (EMCO-TEST, Austria). The microhardness measurements were carried out using the Vickers method in accordance with ASTM E384-17 [22]. The test consisted in performing three measurements on each of the samples by means of a pyramidal diamond indenter with a square base and an apex angle of 136° . Then the indentation diagonals were measured and the hardness value was calculated. The results were presented as arithmetic means. A TRB tribometer (CSM Instruments, Switzerland) in the ball-on-disc configuration was used to determine the tribological properties of the coatings. The sliding pair consisted of a stationary ball with a diameter of 6 mm and hardness 62 ± 2 HRC, made of steel 100Cr6, pressed against a rotating disc made of steel 12Ch18N10T respectively uncoated and spray coated with 500 μm thick Al_2O_3 and ZrO_2 coatings. It was decided to measure the tribological properties of 500 μm thick coatings since they were characterized by similar roughness. The experiment was carried out under air at the temperature of 20°C and the humidity of 34% in technically dry friction conditions. The contact load amounted to 10 N and the rubbing speed to 0.1 m/s. The cycle time was 60 min. The samples were subjected to testing immediately after the spraying process, without a preliminary running-in. Before the measurement the samples had been cleaned with acetone. The tests were carried out in accordance with ASTM G99-95a [23]. The tribological tests were repeated three times and the results were presented as arithmetic means. The surface roughness and the volume of the removed coating material were determined on the basis the cross section of the wear track on the sample's surface by means of the contact profiler of a model 130 profilometer. The structure of the coating groove and the wear spots on the ball were examined under a Neophot-21 optical inverted microscope. In order to determine the adhesive characteristics of the coatings the scratching method a Micro Scratch Tester (CSM Instruments, Needham, Massachusetts, USA) was used. Scratch testing was carried out at a maximum load of 30 N, the normal load change on the sample was 29.99 N min^{-1} , the movement of the indenter was 9.63 mm min^{-1} , the length of the scratch was 10 mm and the radius of curvature of the tip was 100 μm .

4. Results

4.1. Microstructure examinations

Images of the surface and cross-section of the coatings are shown in figures 2 and 3, respectively. Data on the change in roughness (the arithmetic mean deviation of the R_a profile) of the coatings are presented in table 1. An analysis of the surface morphology shows that an increase in layer thickness leads to a decrease in surface roughness, which is characteristic of detonation spraying. An elemental point analysis of the cross section of the coatings was carried out. The elemental composition of the Al_2O_3 and ZrO_2 coatings is shown in figure 4. As one can see, no gas mixture components are present on the coating surface during the detonation spraying process.

It follows from the diffraction patterns shown in figures 5 and 6 that the coatings contain a cubic space group Fd-3m, a hexagonal space group R-3c for Al_2O_3 and a cubic space group Fd-3m for ZrO_2 . Alumina can exist in several crystalline modifications: α - Al_2O_3 , γ - Al_2O_3 , δ - Al_2O_3 , θ - Al_2O_3 , χ - Al_2O_3 , etc. At high temperatures all the phases usually pass into the most stable hexagonal α -phase. In this crystalline state the alumina has maximum density and hardness. Pure zirconium oxide at room temperature exists in a monoclinic form and when heated above 800°C – 1000°C , passes into a more dense cubic modification.

X-ray diffractometry reveals that during detonation spraying a phase transformation occurs in the alumina: the initial Al_2O_3 powder consisted of α - Al_2O_3 , whereas the coating consists of γ - Al_2O_3 (66%) and α - Al_2O_3 (34%). The powder's coherent scattering zones are 215 nm in size while in the coating this size amounts to 136 nm for the α phase and to 31 nm for the γ phase. This is rather unexpected since, as mentioned above, the α phase is more stable at high temperatures.

As a hypothesis, one can assume that the phase transition occurs upon the cooling of the particles falling on the substrate. Smaller and hotter particles deform more strongly on impact, whereby the thickness of the splat is small and the latter cools quickly due to intense heat removal to the substrate. Larger and less heated particles deform less, whereby their splats are thicker and so cool slower and the phase transition $\alpha \rightarrow \gamma$ has time to

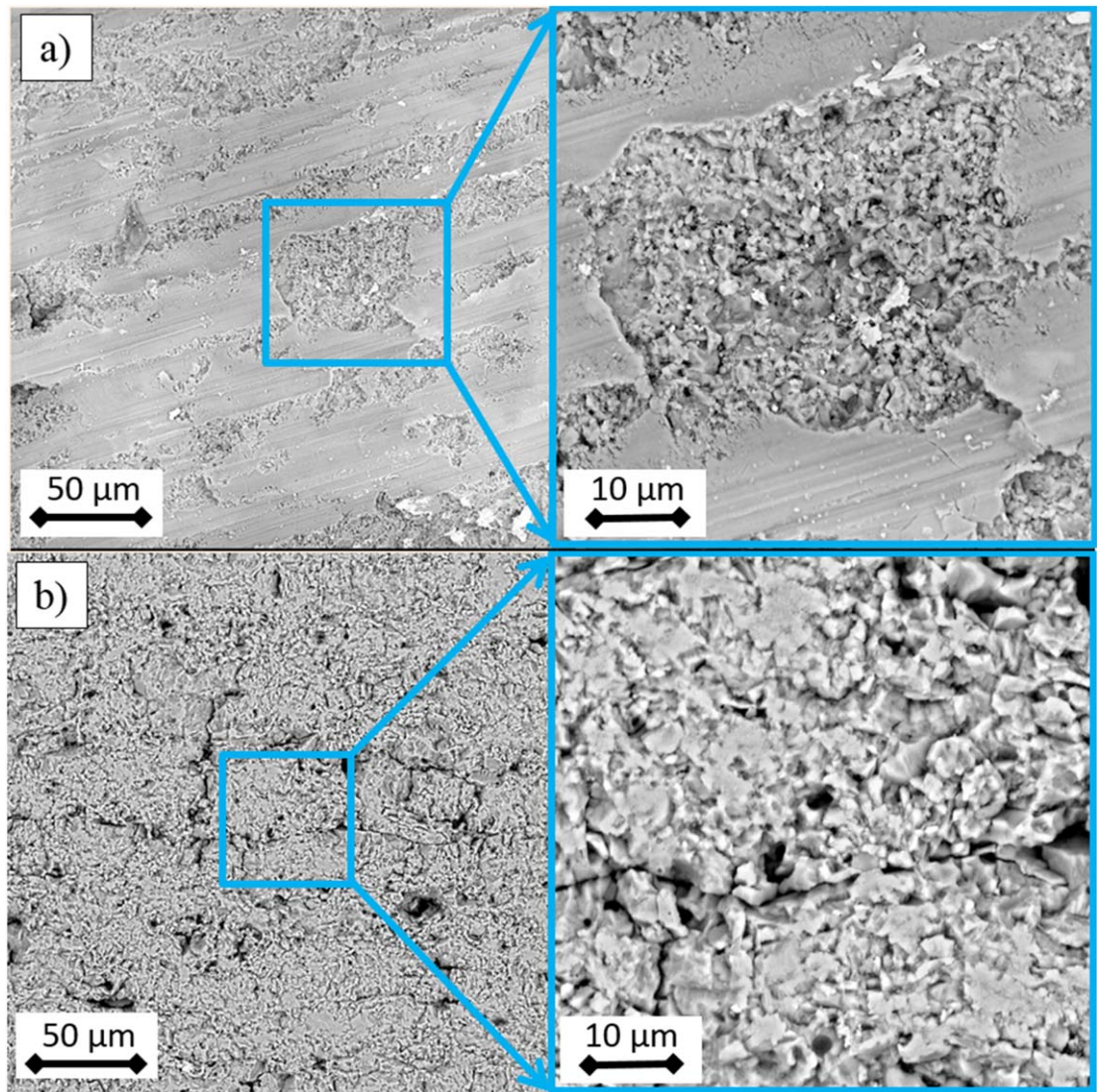


Figure 2. Surface of Al_2O_3 coatings of different thickness: (a) 500 μm , $R_a = 5.10 \mu\text{m}$; (b) 1100 μm , $R_a = 2.20 \mu\text{m}$.

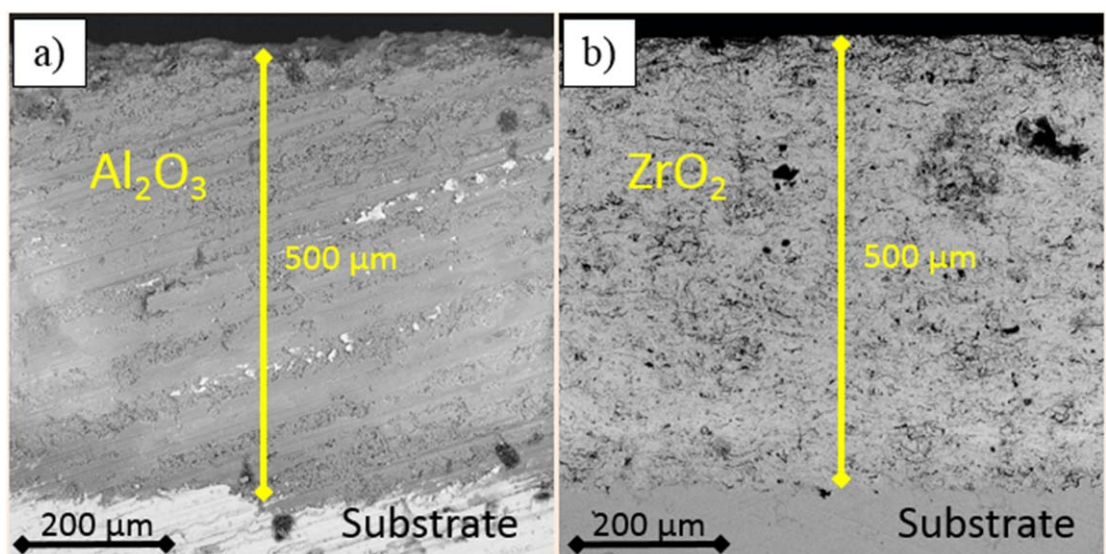


Figure 3. SEM image of cross section of: (a) Al_2O_3 coating and (b) ZrO_2 coating.

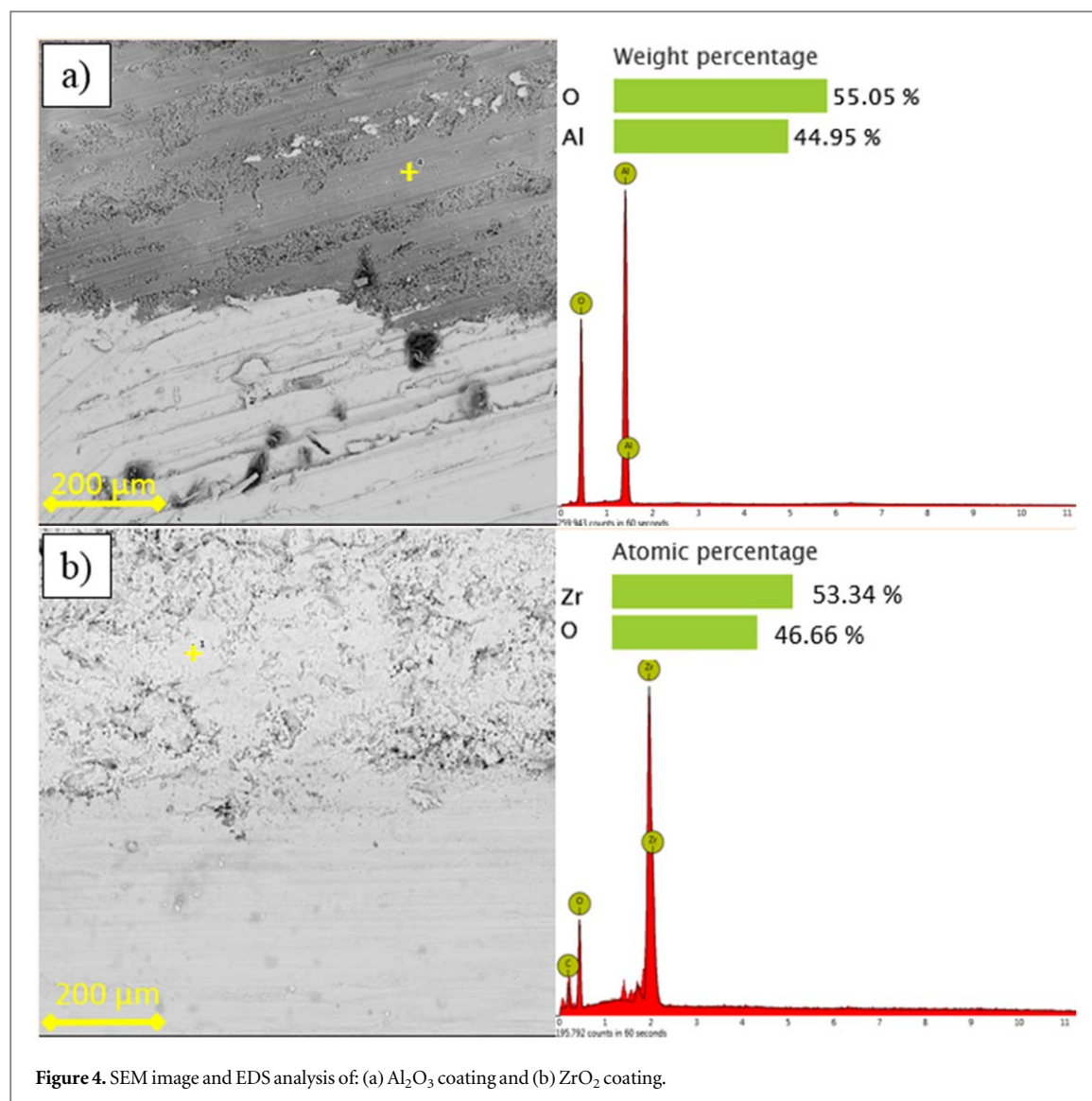


Figure 4. SEM image and EDS analysis of: (a) Al₂O₃ coating and (b) ZrO₂ coating.

occur. Thus, one can conclude that the speed, the temperature and the particle size at the instant of collision with the substrate affect the phase transition as the shape of the splat depends on these parameters.

4.2. Measurements of mechanical properties

The most universal parameter allowing one to evaluate the mechanical properties of coatings quite quickly is microhardness. The results of microhardness measurements for the Al₂O₃ and ZrO₂ coatings are presented in figure 7. Microhardness amounted to 3580 MPa for the uncoated substrate, 7860 MPa for the detonation sprayed 250 μm thick Al₂O₃ coating, 10700 MPa for the 500 μm thick coating and 11080 MPa for the 1100 μm thick coating, i.e. it increased 3-fold relative to that of the uncoated substrate. As the thickness of the Al₂O₃ coating increases, its microhardness significantly improves. After detonation spraying with ZrO₂ powder, the microhardness amounted to 8750 MPa, 8650 MPa and 7100 MPa at a thickness of respectively 250 μm, 500 μm and 1000 μm. This means that for the thickest ZrO₂ coating nearly a two-fold higher microhardness value than that of the uncoated steel was registered. The increase in the thickness of the ZrO₂ coating resulted in slightly worse mechanical properties of the latter.

4.3. Tests of adhesion and tribological properties

One of the main factors determining the quality of the coating is adhesion. Figures 8 and 9 shows the results of testing the adhesive strength of coatings by scratch testing. The moment of adhesion or cohesive destruction of the coating was recorded after visual testing using an optical microscope equipped with a digital camera, as well as by changing two parameters: acoustic emission and friction force. It should be noted that not all recorded parameters, associated with the destruction of the coating, describes the actual adhesion of the coating to the substrate. Recording of various parameters in the testing process makes it possible to determine different stages

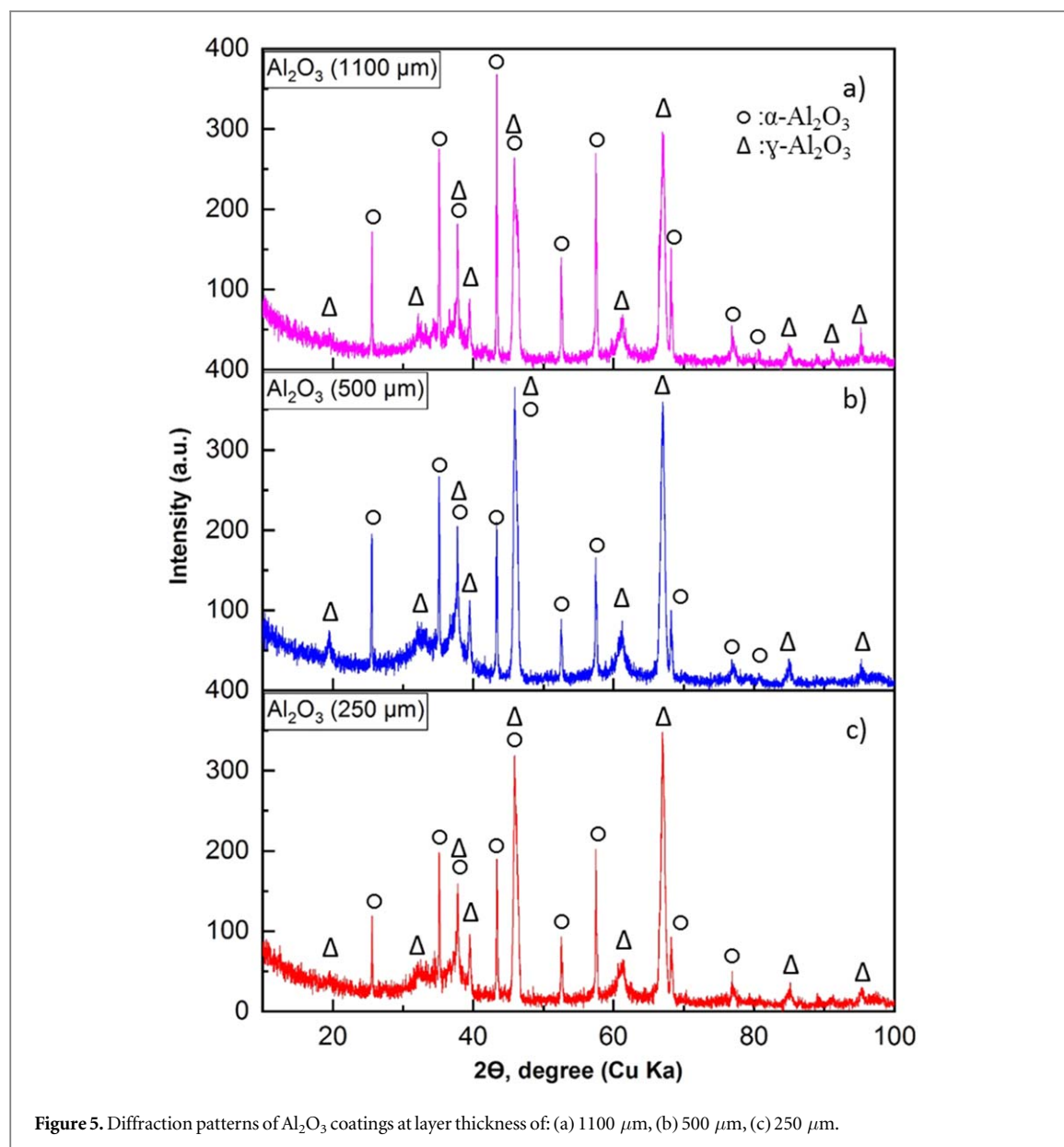


Figure 5. Diffraction patterns of Al_2O_3 coatings at layer thickness of: (a) 1100 μm , (b) 500 μm , (c) 250 μm .

of coating destruction. Thus, L_{C1} indicate the moment of the appearance of the first crack, L_{C2} - peeling of the coating regions, L_{C3} - plastic abrasion of the coating to the substrate [24]. We can estimate the intensity of cracks formation and their development in the sample during scratching by the type of change in the amplitude of acoustic emission (AE). It was observed that for Al_2O_3 coatings obtained with a detonation barrel filling volume of 71%, the first crack was formed at a load of $L_{C1} = 4.57$ N. Then, the process continues with a certain frequency. A corresponding peak of acoustic emission accompanies each formation of a chevron crack (figures 8(a) and (b)). The partial abrasion of the coating to the substrate was estimated based on a sharp change in the intensity of growth of the friction force. This took place at a load of $L_{C3} = 25.24$ N, which was also confirmed by visual observations that showed a change in the color of the sample material at the bottom of the scratch (figures 8(c) and (d)). This value of L_{C3} indicated a high adhesive strength of the coatings to the substrate.

Figures 10 and 11 show the scratch test results for a ZrO_2 coating.

It was observed that for ZrO_2 coatings obtained with a detonation barrel filling volume of 81%, the first crack was formed at a load of $L_{C1} = 11.06$ N. Then, the process continued with a certain frequency. A corresponding peak of acoustic emission accompanies each formation of a chevron crack (figures 10(a) and (b)). The partial abrasion of the coating to the substrate was estimated based on a sharp change in the intensity of the growth of the friction force. This took place at a load of $L_{C3} = 26.28$ N, which was also confirmed by visual observations that showed a change in the color of the sample material at the bottom of the scratch (figures 10(c) and (d)). This value of L_{C3} indicated a high adhesive strength of the coatings to the substrate.

Figure 12 shows the kinematic friction coefficient-time dependence for the Al_2O_3 coating and the ZrO_2 coating, determined by means of a ball-on-disc tribometer. The tests were carried out in dry friction conditions.

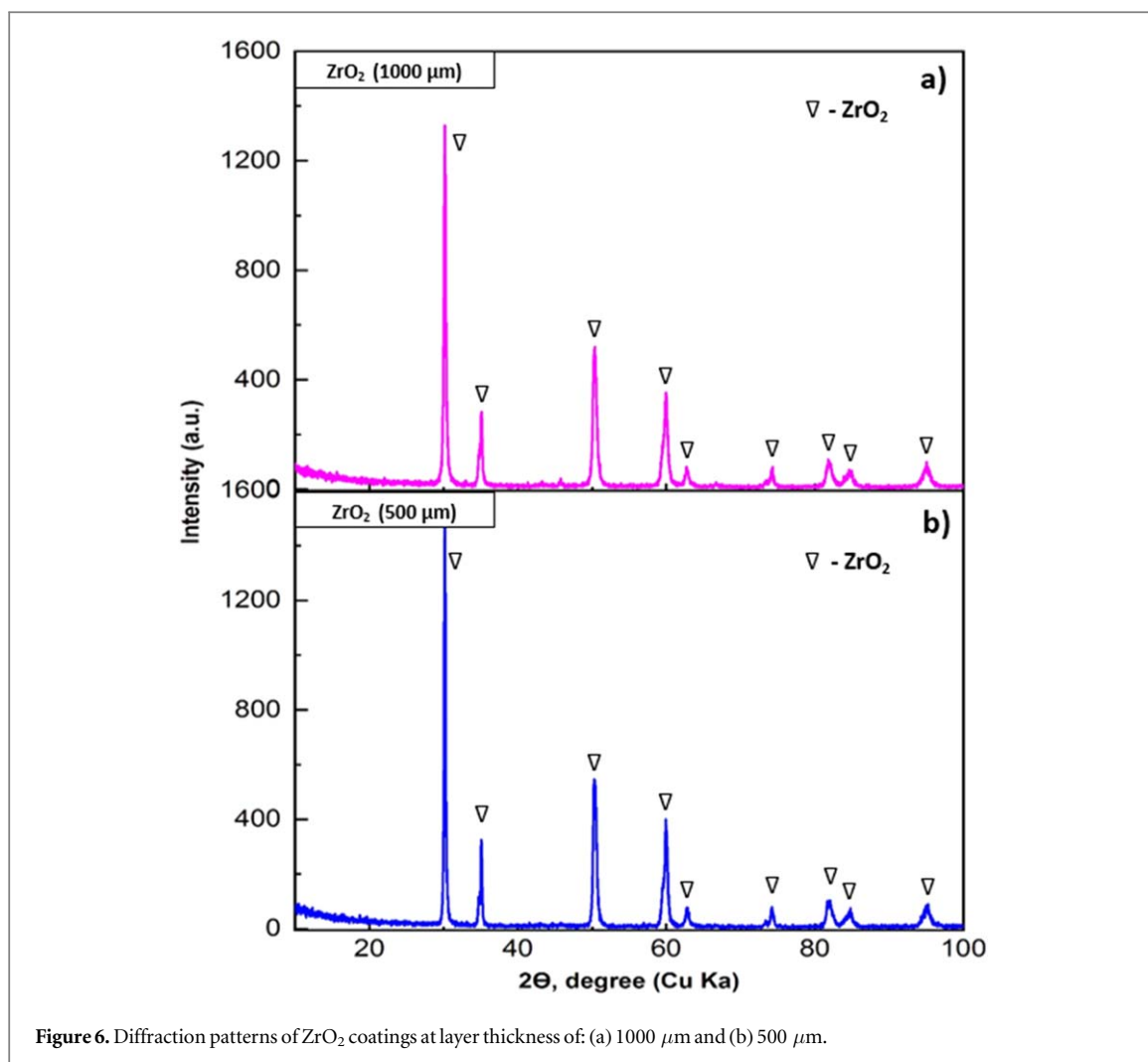


Figure 6. Diffraction patterns of ZrO₂ coatings at layer thickness of: (a) 1000 μm and (b) 500 μm.

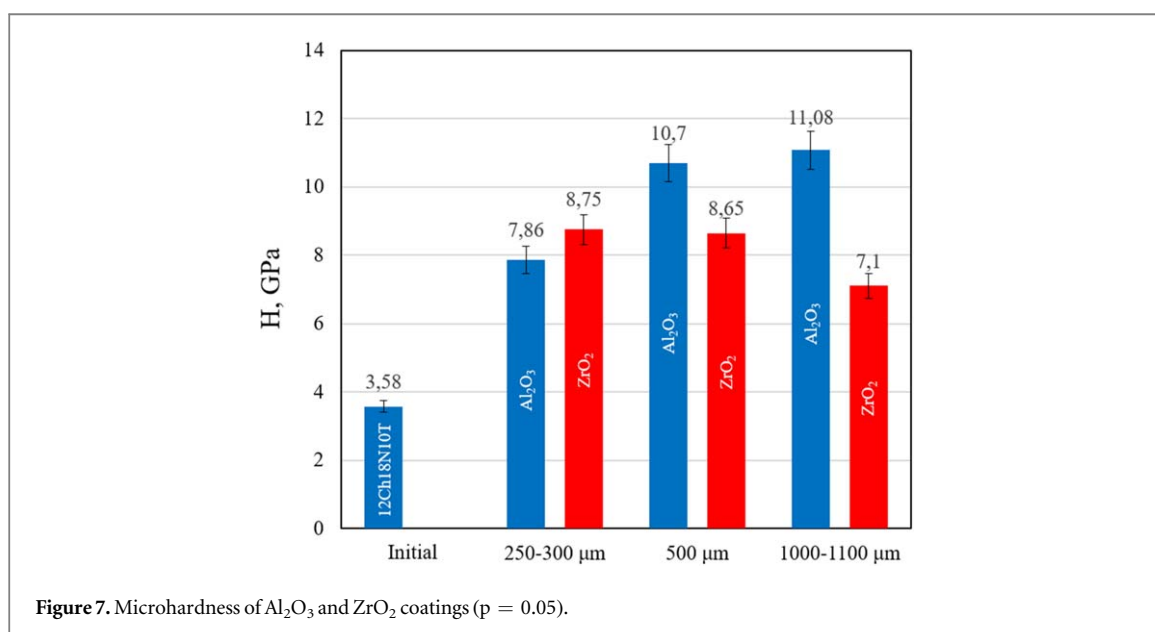


Figure 7. Microhardness of Al₂O₃ and ZrO₂ coatings ($p = 0.05$).

The average value of the friction coefficient of the substrate amounted to 0.669. After the deposition of respectively aluminium oxide and zirconium oxide the average friction coefficient value decreased to 0.603 and 0.330, respectively. Thus the friction coefficient of the zirconium oxide coating is lower than that of the alumina coating. In the case of uncoated steel 12Ch18N10T, the friction process proceeded in an unstable manner along

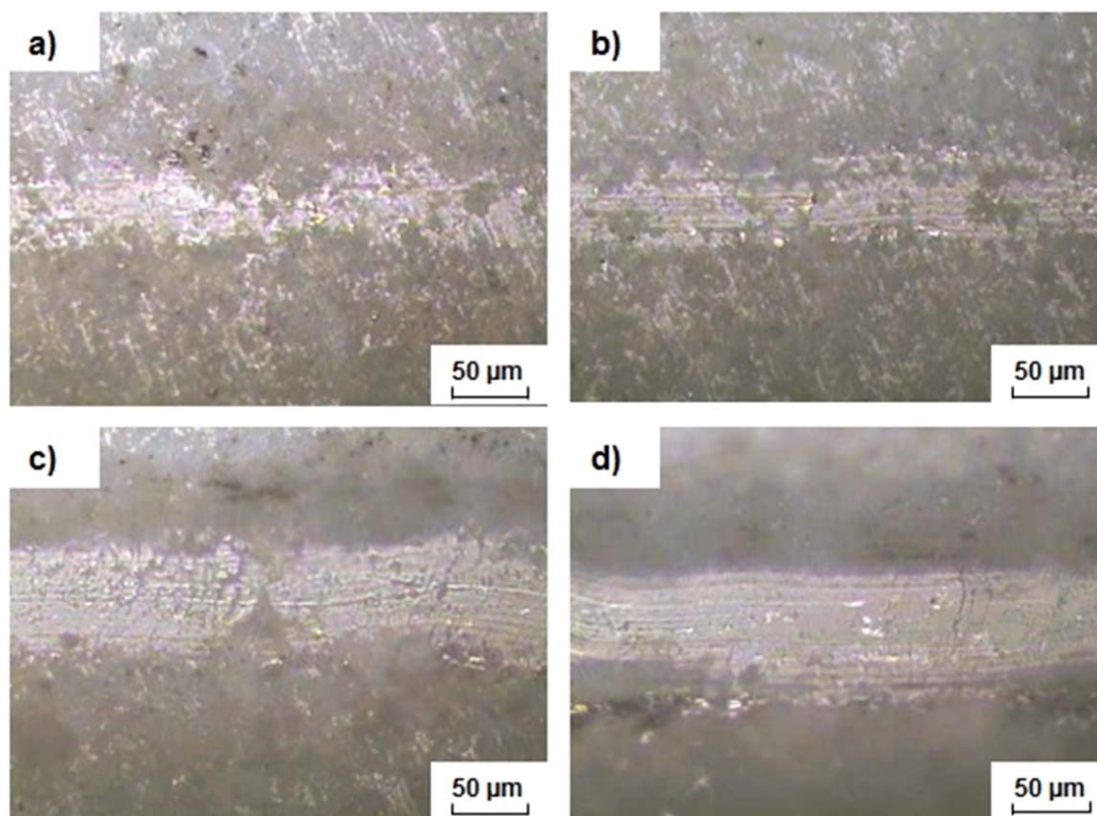


Figure 8. Microphotograph of the area in contact with the diamond indenter during scratch testing of Al_2O_3 coating.

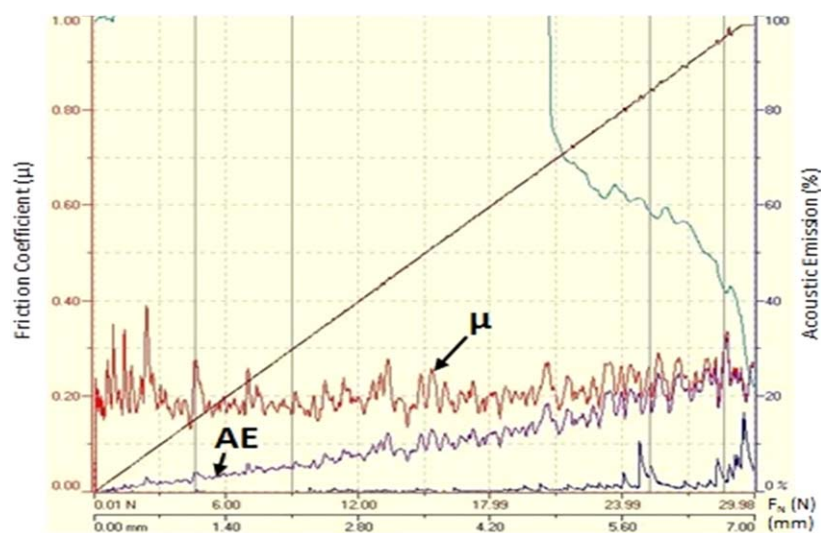


Figure 9. Correlation between the coefficient of friction under load and acoustic emission signal used in scratch testing of Al_2O_3 coating.

the whole friction path. In the initial stage of running-in the friction coefficient value would rapidly increase and after 30 min it would stabilize and decrease. In the case of the oxide coatings, the friction process was much more stable. In both cases, the course of changes in the kinetic friction coefficient had a logarithmic character. A slightly less rapid increase in the friction coefficient was registered in the case of the ZrO_2 coating. The depth profiles of the wear traces are shown in figure 13. As one can see, for the Al_2O_3 coating ($500\text{ }\mu\text{m}$) and the ZrO_2 coating ($500\text{ }\mu\text{m}$) the wear trace depth is minimal. The maximum wear was registered for the uncoated substrate (12Ch18H10T). The cross-sectional area of the wear trace for the uncoated substrate was 0.308 mm^2 . Much

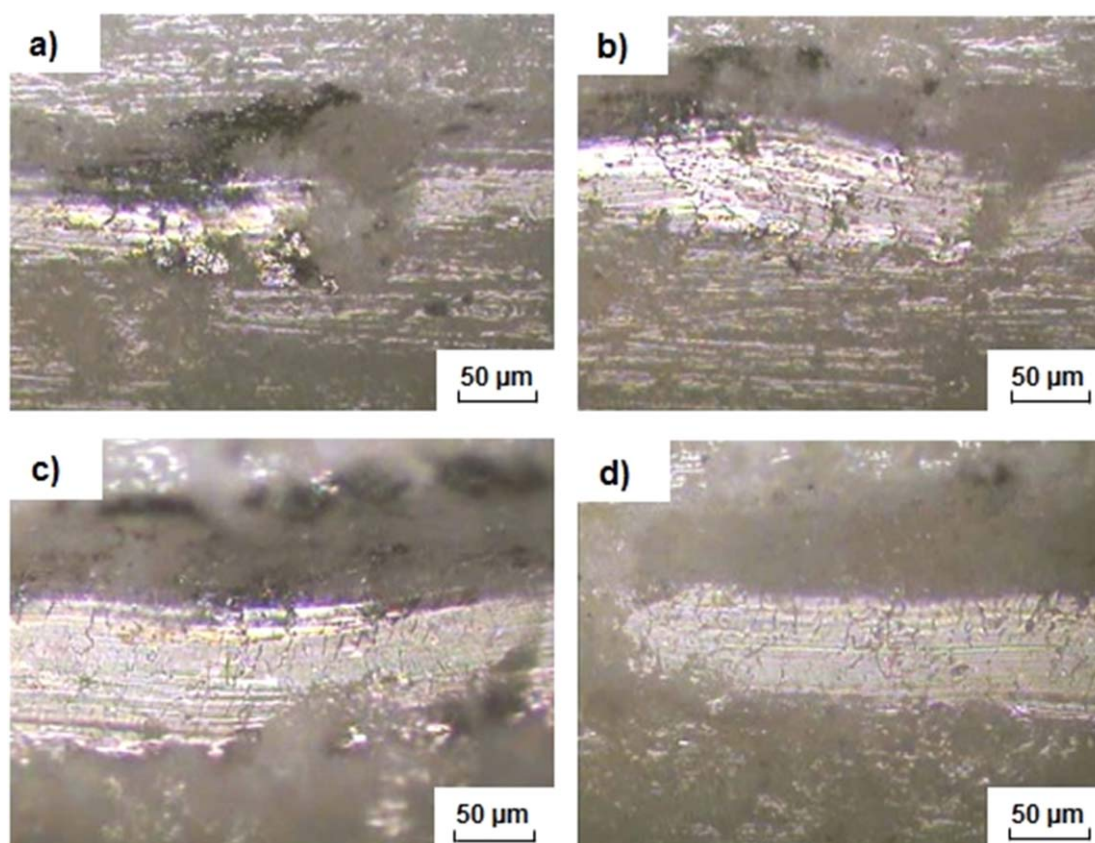


Figure 10. Microphotograph of the area in contact with the diamond indenter during scratch testing of ZrO₂ coating.

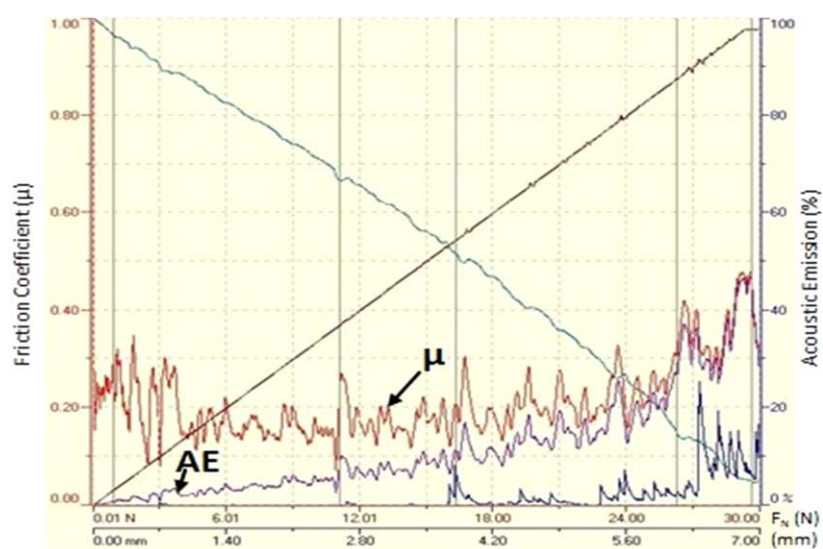
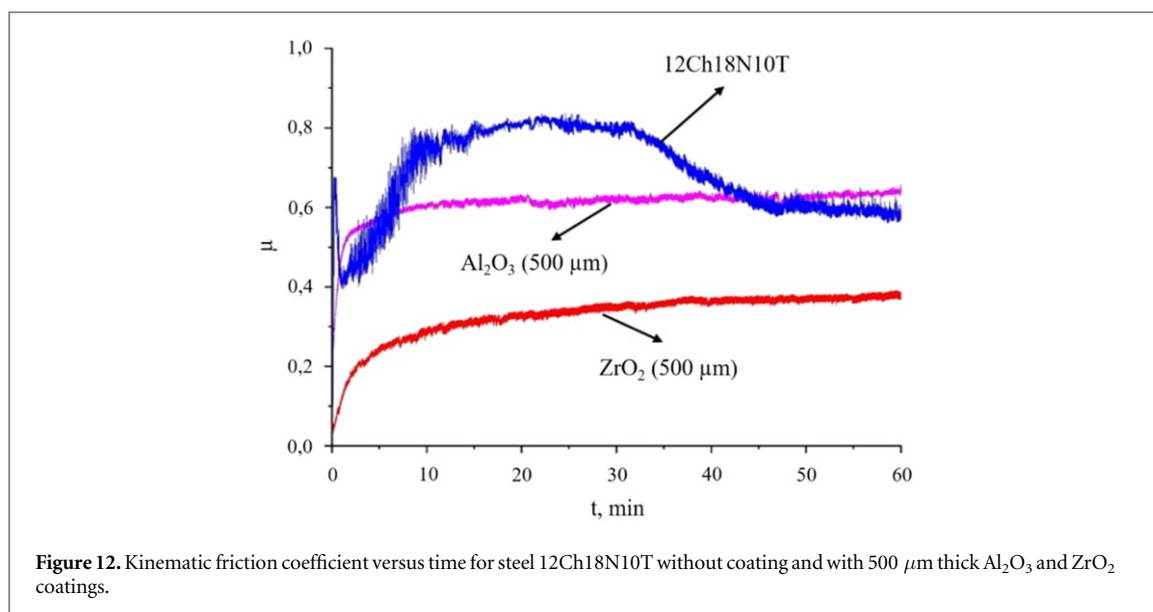


Figure 11. Correlation between the coefficient of friction under load and acoustic emission signal used in scratch testing of ZrO₂ coating.



lower values were recorded for the Al_2O_3 and ZrO_2 coatings. The cross-sectional areas of the wear traces in this case were 0.249 mm^2 and 0.183 mm^2 , respectively.

5. Conclusions

The results of experimental investigations of the physical and mechanical properties, structure and surface morphology of Al_2O_3 - and ZrO_2 -based coatings produced by detonation spraying have been presented. The following conclusions emerge from the presented research:

- 1) A two-phase structure with cubic and hexagonal crystal lattices forms during the deposition of Al_2O_3 , while a single-phase structure with a cubic crystal lattice forms during the deposition of ZrO_2 .
- 2) No components of the gas mixture used in the detonation process were found to be present on the surface of steel 12Ch18N10T after the deposition of Al_2O_3 and ZrO_2 .
- 3) Profilometry tests showed a decrease in the roughness of the Al_2O_3 coating as its thickness was increased in the range of 250–1100 μm . The reverse was observed for the ZrO_2 coating: its roughness increased with its thickness.
- 4) Tests of the mechanical properties of the coatings showed that the microhardness of the Al_2O_3 coatings significantly improved as their thickness was increased in the range of 250–1100 μm . For the thickest coating the increase in microhardness was threefold in comparison with uncoated steel 12Ch18N10T. The reverse tendency was observed for the ZrO_2 coatings. For the thinnest ZrO_2 coating a twofold increase in microhardness in comparison with the uncoated steel was registered. Among the tested 250 μm thick coatings the ZrO_2 coating exhibited the best mechanical properties.
- 5) The ZrO_2 coatings were found to be characterized by the best tribological properties. The registered mean value of the kinetic friction coefficient for the 500 μm thick ZrO_2 coating was nearly by half lower than the value registered for the Al_2O_3 coating of the same thickness. A significant improvement in the tribological properties of the Al_2O_3 and ZrO_2 coatings relative to uncoated steel 12Ch18N10T was noted.
- 6) The wear traces left after tribological testing indicate that the ZrO_2 coatings have better resistance to abrasive wear than the Al_2O_3 coatings.

The experimental research results represent a new step in the development of Al_2O_3 - and ZrO_2 -based protective coatings whose structural and phase characteristics improve the performance of various products operating at high temperatures, loads and wear rates. The results indicate that the performance characteristics of the detonation sprayed ZrO_2 coatings are better than those of the Al_2O_3 coatings produced using the same technique. Coatings based on aluminum and zirconium oxide obtained with the detonation method can provide excellent protection against chemical and tribological wear of the structural elements of gas turbine engines of combined heat and power plants, in particular turbine shafts, discs and rotors. Currently, the authors conduct

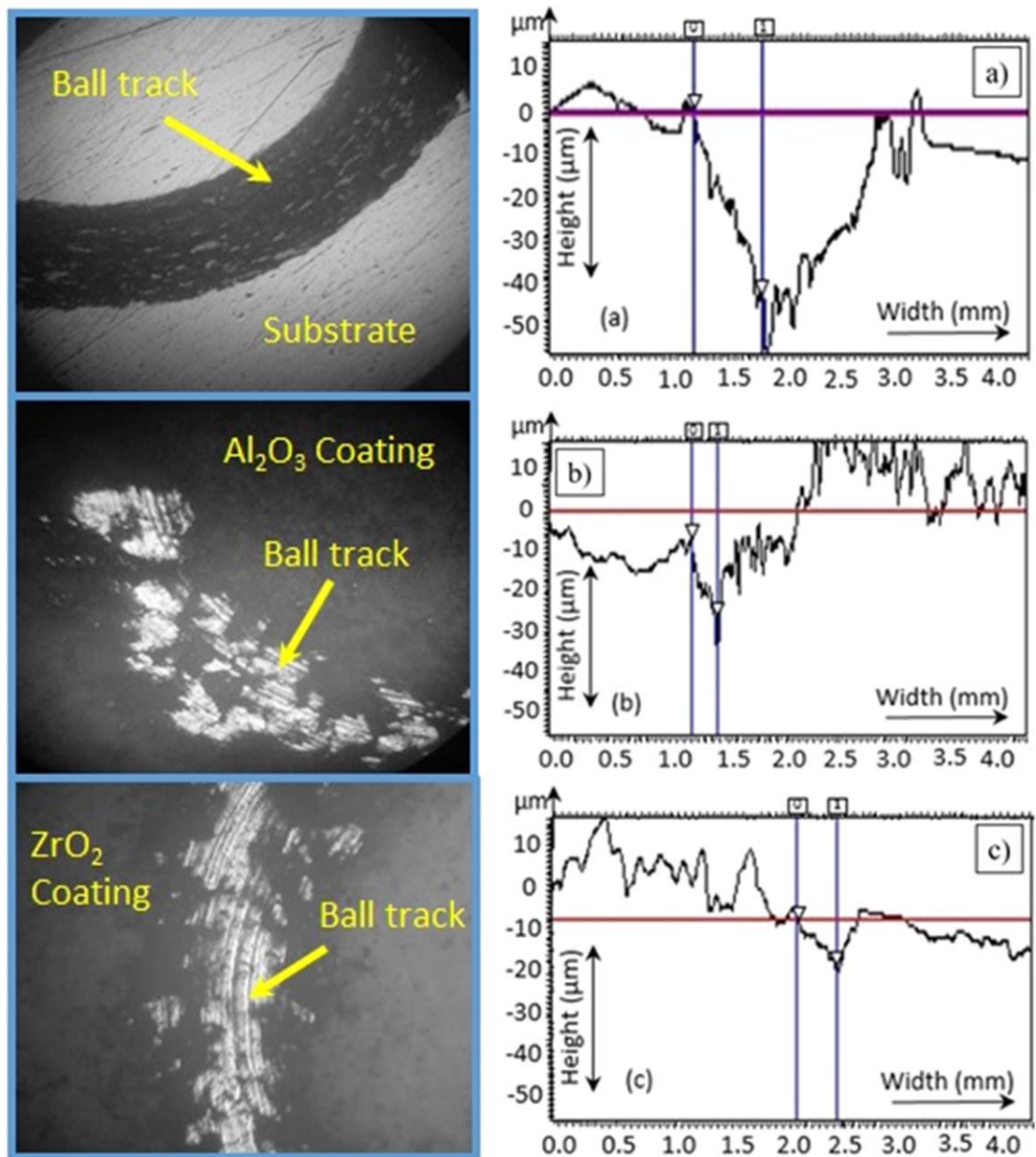


Figure 13. Depth profiles of wear traces in: (a) steel 12Ch18H10T, b) Al_2O_3 coating and (c) ZrO_2 coating.

advanced studies of gradient coatings obtained as a result of changes in detonation parameters. These coatings are characterized by improved performance properties. In the future, the authors intend to carry out structural, phase, physicochemical and tribological tests of oxide coatings obtained with the detonation method for different levels of gas filling in the combustion chamber of the detonation gun. There are also plans to test the resistance of oxide coatings to thermal radiation and corrosion.

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