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AN ANALYSIS OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF Zr/Zr_2N MULTILAYER COATINGS

ANALIZA WŁAŚCIWOŚCI MECHANICZNYCH I TRIBOLOGICZNYCH POWŁOK WIELOWARSTWOWYCH Zr/Zr_2N

Key words: multilayer coatings, PVD, nanohardness, friction, wear.

Abstract: The paper presents the analysis of effect of the Zr to Zr_2N layer thickness ratios of 1:1, 1:2, and 1:4 on the mechanical properties of Zr /Zr_2N multilayer coatings. With the increase in the amount of the ceramic phase in the multilayer coating, an increase in hardness from 11 to 14.5 GPa and Young's modulus from 158 to 211 GPa was observed. This was accompanied by improved scratch resistance as critical load L_{C2} raises from 17 to over 30 N. A ratio increase from 1:1 to 1:2 caused a 3-fold improvement in the coating wear resistance, which was also accompanied by a change in the wear mechanism. However, there was no further improvement of this parameter with a ratio increase to 1:4. Microscopic analysis of wear tracks, using the TEM technique revealed that the Zr metal layers change the direction of crack propagation. This phenomenon results in the improved fracture resistance of multilayer coatings compared to single coatings. Such a mechanism was even observed for the 1:4 coating with the thinnest 58 nm Zr layers.

Słowa kluczowe: powłoki wielowarstwowe, PVD, nanotwardość, tarcie, zużycie.

Streszczenie: W pracy przedstawiono analizę wpływu stosunku grubości warstw Zr i Zr_2N 1:1, 1:2 i 1:4 na właściwości mechaniczne powłok wielowarstwowych Zr/Zr_2N. Wraz ze wzrostem udziału fazy ceramicznej w powłoce wielowarstwowej obserwowano wzrost jej twardości z 11 do 14,5 GPa i modułu Younga ze 158 do 211 GPa. Towarzyszyła temu także poprawa odporności na zarysowanie, o czym świadczy wzrost wartości obciążenia krytycznego L_{C2} z 17 do ponad 30 N. Wzrost stosunku z 1:1 do 1:2 powodował 3-krotną poprawę odporności na zużycie powłoki, czemu towarzyszyła także zmiana mechanizmu zużywania. Natomiast nie następowała dalsza poprawa tej cechy przy wzroście stosunku do 1:4. Analizy mikroskopowe torów tarcia przy użyciu techniki TEM wykazały, że warstwy metali Zr powodują zmianę kierunku propagacji pęknięć, co przekłada się na poprawę odporności na pękanie powłok wielowarstwowych w stosunku do powłok pojedynczych. Mechanizm taki obserwowano nawet dla powłoki 1:4, dla której grubość warstwy Zr była najmniejsza i wynosiła 58 nm.

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INTRODUCTION

The growing requirements for energy efficiency and durability of machines, engines, and other technical facilities demand continuous efforts to reduce the friction forces and wear of friction nodes. In tribology, two directions are developing to meet these requirements. Newer lubricants are introducing; however, they cause many problems related to the ecology of their production, collection after the operation process, and finally with their utilization. The second solution dominates where the use of oils is risky or impossible is the surface modification through various technologies in the area of surface engineering, so that the friction nodes can work in dry sliding conditions **[L. 1]**. Thin anti-wear coatings have been using in this field for a long time and, in recent years, mainly applied by physical techniques from the gas phase – PVD **[L. 2]**. The development of these technologies allowed the coatings deposition at room temperature; hence, there are no restrictions as to the extent of the material capabilities of the coating itself as well as the substrates on which the coatings can be deposited. In mechanical applications, the main problem is to protect the coating-substrate system itself against large deformations. These deformations, due to external loads, lead in a case of soft substrates, e.g., polymeric, soft metals, such as titanium alloys and soft steels, to the same deformation of the coating. On the other hand, the necessity of limiting wear causes that very hard coatings, ceramic as carbides or nitrides, of transition metals or carbon coatings, which are characterized by low resistance to cracking, are usually deposited. For such systems, large contact stresses lead to the fracture of coatings and, as a consequence, to their fragmentation which causes the wear acceleration of tribological systems when wear products remain in the friction zone.

Hence there is a strong need to produce coatings simultaniously with high hardness and with high fracture resistance. Single coatings, like TiN, CrN, TiC, and DLC cannot handle these requirements. Their hardness could be up to 40GPa, but the critical stress intensity factor K_C usually does not exceed $5MPa \cdot m^{0.5}$ **[L. 3]**.

The idea of a solution of this problem for engineers could be a shell where the nacre is composed of crystallites in the form of thin aragonite tablets with 15 μm diameters and 0.5 μm thicknesses. The structure also contains thin layers of organic matter not thicker than 0.3 μm, gluing aragonite crystallites **[L. 4]**. Due to this construction, the aragonite can slightly slide apart when predators are hitting the shell, without breaking the shell structure. The ceramic-metal multilayer coatings have similar structures **[L. 5]**. In such coatings, metal and ceramic layers with thicknesses from a few to a few hundred nanometres are arranged alternately. This causes that it is possible to obtain properties unattainable by either single metal or ceramic coatings. An appropriate selection of materials forms a structure where thin successive layers and the typical crystalline columnar structure for PVD coatings are proportional to the thickness of these layers. This fine grained microstructure of thin layers leads to hardness increase. In addition, properly selected crystal lattices, with not too big mismatch of their parameters, give coherent or semi-coherent interlayer boundaries. Such a character of these boundaries makes them an energy barrier for dislocations motion, especially when passing from metal to ceramic layers. A large number of dislocations in metal layers at the border with ceramic ones also strengthen the metal layers. In addition, for very thin layers with a small difference in lattice parameters, an additional stress field arise due to the mismatch of the crystal lattice, while these differences cannot be too high, because the resulting interfacial boundaries are incoherent and then this mechanism stops working. The latest literature presents the results of tests for various material systems Ti/TiN **[L. 6]**, Al/AlN **[L. 7]**, Cr/CrN **[L. 8]**, and others. The relations between mechanical properties of such multilayers and their period are mainly sought **[L. 8**, **9]**. The period is the sum of the thicknesses of two successive layers, i.e. metal and ceramic. Previous works of authors allowed us to determine the optimal value of multilayer periods and to analyse the possibilities of improving crack resistance by introducing metal layers **[L. 9**, **10]**. In this work, the research program has been undertaken to analyse the effect of the thickness ratio of metal and ceramic layers in a multilayer coating. What should be a thickness of metal to be an effective barrier for crack closing? On the other hand, thick metal layers can significantly reduce the hardness of the entire coating, and plastic deformations of the metal cause that they will no longer provide adequate support for the ceramic layers, which can easily crack. The analysis was carried out for Zr/Zr_2N material system, for which coatings with different thickness ratios Zr and Zr_2N were produced with the same period. Their mechanical and tribological properties were compared.

MATERIALS AND RESEARCH METHODOLOGY

The tested Zr/Zr_2N multilayer coatings along with their structure and the thicknesses of individual layers are listed in **Table 1**. They were applied by magnetron sputtering in Joanneum Research – Institute for Surface Technologies and Photonics, Niklasdorf in Austria. In order to apply multilayer coatings, the atmosphere in the chamber was changed and the mixture of nitrogen and argon gases was the Zr_2N (ceramic) nitride layers, and argon was applied for Zr metal layers. The high purity Zr target was sputtered and the deposition temperature did not exceed 40°C. The thickness of individual layers was controlled by controlling the time of their deposition in an appropriate atmosphere, after determining the

rate of their growth for thick coatings. All coatings were deposited on X5CrNi18-10 austenitic stainless steel substrate. Steel surfaces, before deposition, were polished then degreased, washed, and placed in a vacuum chamber (Leybold Univex 450). The low roughness of the substrates, in the case of multilayer coatings, where successive layers have small thicknesses, is very important, because, during PVD processes, the number of defects depends on substrate topography in a significant way. During the deposition, substrates were mounted on a rotating table, which provide uniformity of coatings on the whole surface.

Table 1. Tested coatings
Tabela 1. Badane powłoki

Coating designation	Total thickness [nm]:	Coating period Λ [nm]	Zr layer thickness [nm]	Zr_2N layer thickness [nm]
8x Zr/Zr_2N r1:1	~2320	~290	~145	~145
8x Zr/Zr_2N r1:2	~2370	~297	~99	~198
8x Zr/Zr_2N r1:4	~2300	~288	~58	~231

Transmission electron microscopy (TEM) and Philips CM20 200 kV and JEOL EX4000 (400 kV) microscopes were used to analyse the microstructure of the coatings. Thin foils for observation were prepared using the FIB (focus ion beam) method. Nanohardness of the coatings was measured by instrumental indentation **[L. 11]** using NHT device manufactured by CSM Instruments. A diamond of Berkovich's geometry was used with 2 and 5 mN maximum loads. The lower load corresponded to a maximum penetration depth of up to 100 nm, i.e. below 10% of the coating thickness, which ensured that the steel substrates have no influence on the results of experiments. For each parameter, at least 6 indentations were made and the values presented are average values. Adhesion of coatings to substrates was measured using scratch tests **[L. 12]** using Rockwell C with 200 μm tip radius diamond indenter. The tests were performed within 0–30 N normal load range along 5 mm scratch length. In order to precisely determine the load that caused cohesive cracks, tests were also carried out up to a load of 2 N. As a result of the tests, critical load values L_{C1} and L_{C2} were determined corresponding to the formation of the first cohesive and adhesive cracks, respectively. The analysis was based on microscopic observations of scratches after the test and an acoustic emission signal. The tribological tests were carried out in ball-on-disc geometry, using Al_2O_3 balls, F = 1 N normal load, and n = 20,000 cycle number. Values of wear index were calculated from the following relation **[L. 13]**:

$$W_V = \frac{V}{F \cdot l} \left[\frac{mm^3}{Nm}\right]$$

where V – volume of removed material, l – track length.

The volume of removed material was determined from measurements of friction track profiles using a ProFilm 3D contactless profilometer (Filmetrics USA), from four places, every 90 degrees, on the circumference.

TEST RESULTS

Microstructure observations using TEM microscopy revealed a layered structure of coatings with well separated ceramic and metal layers. **Figure 1** shows TEM images of coatings with the largest amount of the ceramic phase 1–4 ratio. Although the Zr layers have a thickness of only 58 nm, they are clearly visible on the cross-section. The structure of ceramic layers Zr_2N is columnar, which is typical for ceramic coatings applied by magnetron sputtering. The observations confirmed the thickness of individual layers that were assumed at the design stage.

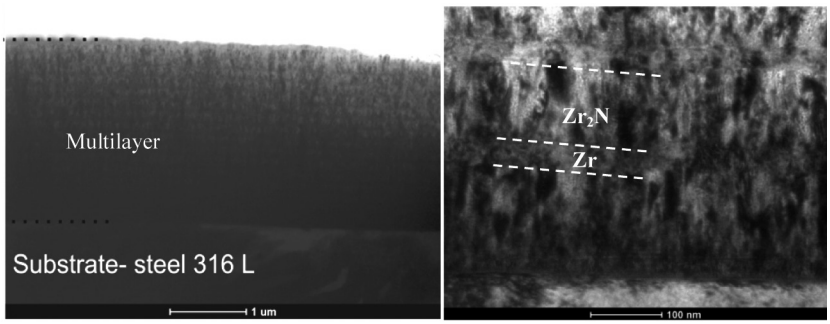

Fig. 1. TEM images of 8x Zr/Zr_2N r1:4 multilayer
Rys. 1. Obrazy TEM powłoki wielowarstwowej 8x Zr/Zr_2N r1:4

The results of indentation tests (nanohardness and elasticity modulus) are presented in **Fig. 2a** and **2b**, respectively. Experiments showed that the nanohardness increases with increasing the thickness of the ceramic layers from 11 to 14.5 GPa (**Fig. 2a**). The values measured for the 5 mN load are about 10% smaller, which is probably related to the fact that deformation reaches the substrate with significantly lower hardness H = 2.5 GPa than the coating. A similar relationship was found for elasticity modulus (**Fig. 2b**) and its increase from 158 to 211 GPa. In both figures, the values calculated form rule of mixtures (RM) were added from tests which were performed previously for 1 μm thick single coatings, for which the hardness and elasticity modulus are H = 5 and 16 GPa, E = 120 and 220 GPa for Zr, and Zr_2N, respectively. The biggest differences between predicted values from RM and experimental results were found for the coating with the highest amount of ceramic phase. This may be due to the finest microstructure of metallic layers with a thickness of 58nm, which can significantly increase the hardness compared to the same single Zr coating, but thicker. Of course, ceramic coatings also have a smaller thickness, whereas, in multilayer coatings, it increases with rising ratio of the ceramic phase to r1-4. Interfaces between layers that restricts movement of dislocations and plastic deformation also play some effect, but in all tested multilayers, their number is the same.

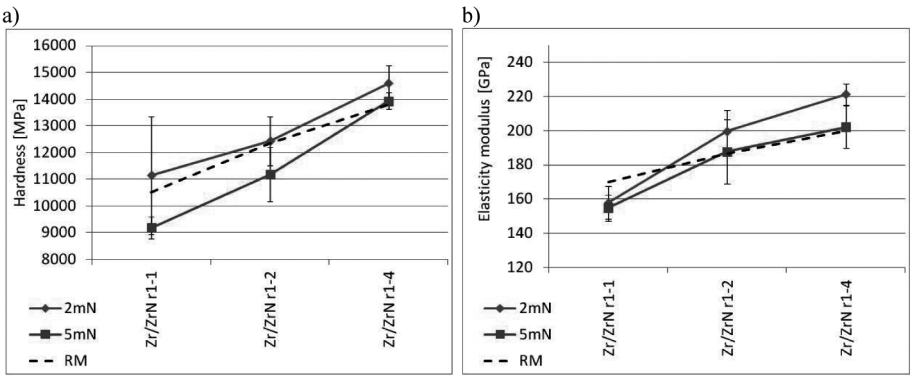

Fig. 2. Indentation test results: a) nanohardness, b) elasticity modulus
Rys. 2. Wyniki testów indentacyjnych: a) nanotwardość, b) moduł sprężystości

An extremely important parameter for the coating-substrate systems, deciding the ability to transfer contact loads, is their fracture resistance and the strength of coating to substrate interface. For 8xZr/Zr_2N r1--1multilayer, with the largest amount of a soft and pliable metallic phase, the first small cracks could be seen at low 0.9 N load (**Fig. 3a**), which is due to the low stiffness of the coating. However, they are not particularly dangerous, because microcracks probably appear only in outer Zr_2N layer. Concave cracks indicate that they were caused by tensile stress behind the indenter. The load increase up to 17.5 N induced coating removals and a small area of substrate exposure (**Fig. 3b**).

The removal of buckled parts due to compressive stress from the whole scratch width occurred at a load of 26.6 N. The remaining, attached coating fragments are highly cracked, and the substrate is visible at the bottom of the groove. Whereas, for the 8xZr/Zr_2N r1-2 multilayer, the first coarse cracks of Zr_2N top layer were formed behind indenter at 1.7 N load (**Fig. 4a**). The change in their character and size took place at 19.5 N (**Fig. 4b**), when the cracks are caused by compressive stresses in front of the indenter. The coating with a 1:2 ratio has a much better adhesion to the substrate than the coating with a 1:1 ratio. The coating was not delaminated, but removed from the substrate due to abrasion at the load L_{C2} – 28.4N (**Fig. 4c**).

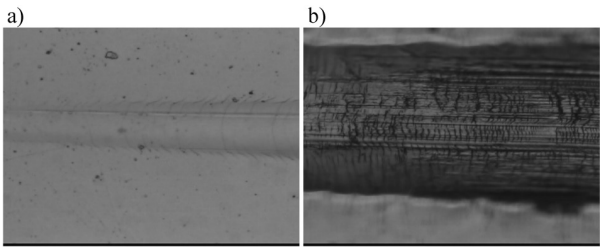

Fig. 3. Images of scratches of Zr/Zr_2N r1-1 coating at characteristic loads: a) 0.9 N, and b) 17.5 N
Rys. 3. Obrazy toru zarysowania powłoki Zr/Zr_2N r1-1 przy obciążeniach charakterystycznych: a) 0,9 N, b) 17,5 N

Thus, the increase in amount of ceramic phase from r1-1 to r1-2 causes an increase in the critical loads. L_{C1} and L_{C2} critical loads for all coatings are listed in **Table 2**. For the 8xZr/Zr_2N r1-4 multilayer, the cohesive

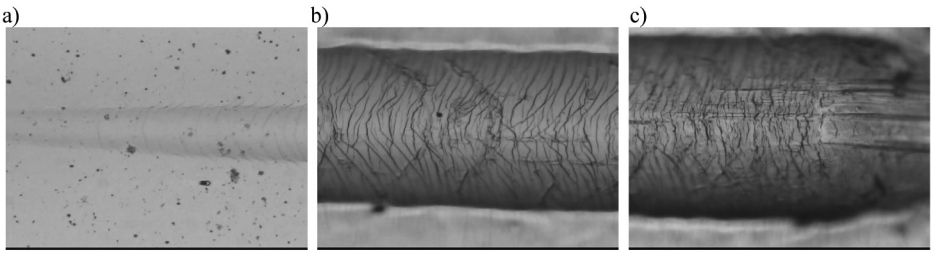

Fig. 4. Images of scratches of Zr/Zr_2N r1-2 coating at characteristic loads: a) 1.7 N, b) 19.5 N, and c) 28.4 N
Rys. 4. Obrazy toru zarysowania powłoki Zr/Zr_2N r1-2 przy obciążeniach charakterystycznych: a) 1,7 N, b) 19,5 N, c) 28,4 N

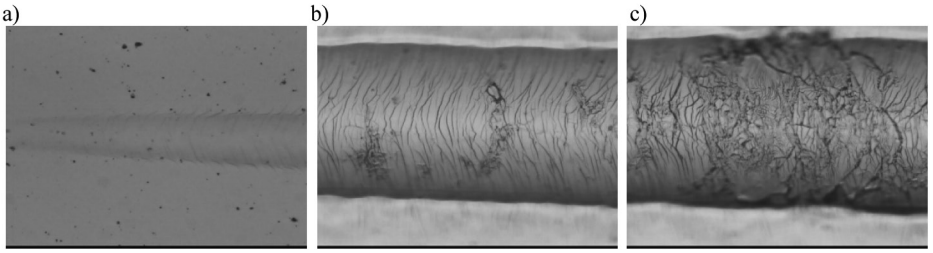

Fig. 5. Images of scratches of Zr/Zr_2N r1-4 coating at characteristic loads: a) 1 N, b) 18.8 N, and c) 23.3 N
Rys. 5. Obrazy toru zarysowania powłoki Zr/Zr_2N r1-4 przy obciążeniach charakterystycznych: a) 1 N, b) 18,8 N, c) 23,3 N

cracks (**Fig. 5a**) appeared at loads of 1N and 18.8 N caused the formation of bigger conformal cracks (**Fig. 5b**). This coating, like coating with 1:2 ratio, is covered with a network of cracks caused by compression and tension before and after the indenter, while the number of cracks due to compressive stress seemed to be smaller. At 23.3 N, the top layer is removed; however, the substrate is not exposed. The maximum load of 30 N does not completely destroy the coating; therefore, Lc2> 30 N is entered in **Table 2**.

Table 2. Critical load values of Zr/Zr_2N multilayers
Tabela 2. Wartości obciążeń krytycznych powłok wielowarstwowych Zr/Zr_2N

Coating	Lc_1	Lc_2
8x Zr/Zr_2N r1-1	0.9±0.3 N	17.5±1.5 N
8x Zr/Zr_2N r1-2	1.7±0.5 N	28.4±2.3 N
8x Zr/Zr_2N r1-4	1±0.3 N	>30 N

The tribological tests showed that the 8xZr/Zr2N r1-1 coating is characterized by the largest wear from all tested coatings. It was about three times larger than the other multilayer coatings. Probably, the low stiffness of the coating, its low hardness, and the occurrence of large plastic deformations observed in scratch tests led to high wear. These plastic deformations of Zr layers increase the deformation of the coating, causing the fracture of Zr_2N ceramics layers and the removal of coating fragments at cyclically repeated contact. **Figure 6a** shows longitudinal grooves in wear track, which may also indicate that wear products remain in the friction zone intensifying the abrasive wear process.

It is also visible in images of the ball at the magnification of 200x, where the fragments of the ceramic having a golden colour are recessed in the surface of the sphere (**Fig. 6d**). The 8xZr/Zr2N r1-2 coating exhibited the best wear resistance among all samples coated with multilayers. The sample profiles were characterized by the occurrence of relatively small

Table 3. Summary of tribological test results of tested coatings
Tabela 3. Zestawienie wyników testów tribologicznych badanych powłok

Coating	Furrow section area [mm^2]	Volume of material removed [mm^3]	Wear indicatory W [mm^3/N*m]	Coefficient of friction
8x Zr/Zr_2N r1-1	$31.1 \cdot 10^{-6}$	$5.9 \cdot 10^{-4}$	$15.5 \pm 2.7 \cdot 10^{-6}$	0.15
8x Zr/Zr_2N r1-2	$10.2 \cdot 10^{-6}$	$1.9 \cdot 10^{-4}$	$5.1 \pm 1.9 \cdot 10^{-6}$	0.1
8x Zr/Zr_2N r1-4	$13.5 \cdot 10^{-6}$	$2.5 \cdot 10^{-4}$	$6.7 \pm 1.6 \cdot 10^{-6}$	0.1

pile-ups, and the maximum groove depth was about 0.4 μm, so the coating was not completely rubbed off (**Fig. 6b**). The size of wear area on the ceramic ball indicates its low wear and the absence of detached large coating fragments in the contact zone (**Fig. 6e**). The last of the tested coatings, 8xZr/Zr_2N r1-4, has only slightly worse wear resistance than the coating with r1-2, and images of the wear track and the surface of the ball indicate a similar nature of wear to the 8xZr/Zr2N r1-2 coating. The average values of the coefficient of friction 0.1–0.15 are similar for all tested samples (**Table 3**).

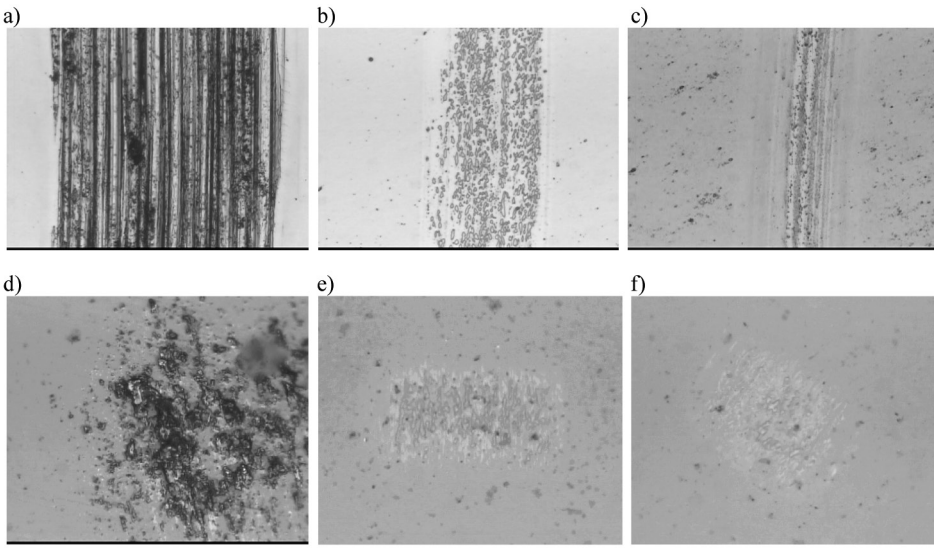

Fig. 6. Wear track images of coatings: a) 8x Zr/Zr_2N r1-1, b) 8x Zr/Zr_2N r1-2, c) 8x Zr/Zr_2N r1-4, and balls after tribological tests: d) 8x Zr/Zr_2N r1-1, e) 8x Zr/Zr_2N r1-2, and f) 8x Zr/Zr_2N r1-4

Rys. 6. Obrazy torów tarcia powłok: a) 8x Zr/Zr_2N r1-1, b) 8x Zr/Zr_2N r1-2, c) 8x Zr/Zr_2N r1-4 oraz kul po testach tribologicznych: d) 8x Zr/Zr_2N r1-1, e) 8x Zr/Zr_2N r1-2, f) 8x Zr/Zr_2N r1-4

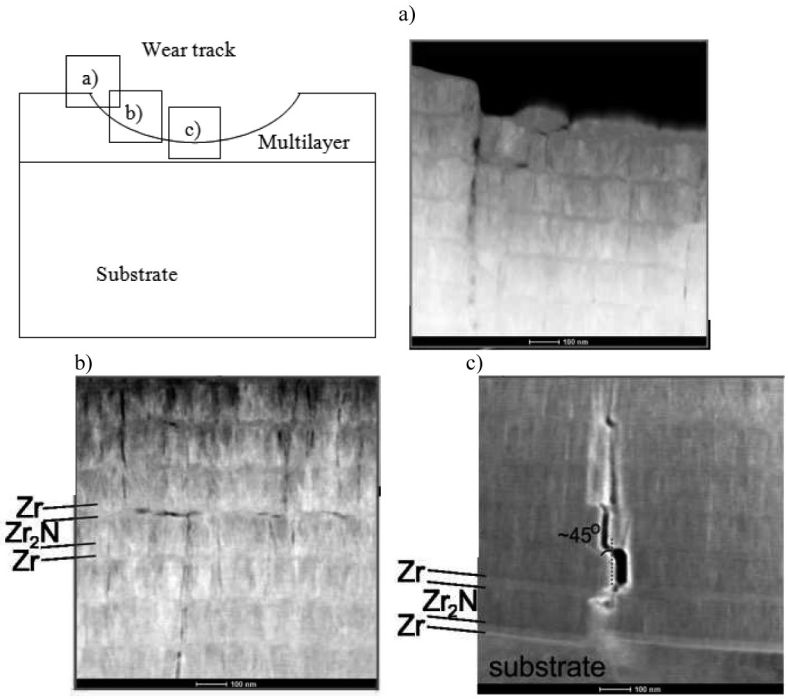

Fig. 7. TEM images of wear track of 8x Zr/Zr_2N r1-4 in areas pointed on the scheme and designated a), b), and c)

Rys. 7. Obrazy TEM toru tarcia powłoki 8x Zr/Zr_2N r1-4 w obszarach zaznaczonych na schemacie oznaczonych a), b) i c)

A slightly higher value for 8xZr/Zr_2N r1-1 coating is probably due to the presence of wear products in the friction zone. TEM images of the wear track of 8xZr/Zr_2N r1-4 coating are shown in **Fig. 7**. Thin foils were made in three characteristic places marked on the schematic drawing of the wear track. In the area (a), i.e. the nearest to edge of track, cracks appear on the surface of the coating and propagate perpendicularly to the coating surface. They result from a high concentration of tensile stresses on the surface in this area. These observations confirm numerical analyses carried out using the finite element method and presented in a previous paper **[L. 14]**.

These cracks are stopped on Zr metal layers after passing one or just a few ceramic layers. Each of the metal layers caused that the increase in the load is necessary to induce crack initiation in the next ceramic layer.

FEM analyses show that it may be the multiple raise of critical load destroying the multilayer coating in relation to a single thick ceramic coating **[L. 14]**. For the area closer to the centre of the wear track (Area b), cracks are formed closer to middle of coating thickness, but they do not propagate completely towards the surface or towards the coating-substrate interface. In the last analysed area (c), the cracks start near the substrate and propagate towards the surface, but, similarly as in the previous cases, they are smaller and smaller on subsequent ceramic layers.

CONCLUSIONS

Multilayer coatings consisting of alternate metal and ceramics layers are increasingly being developed. An appropriate choice of the thickness of these layers can be crucial, considering their mechanical and tribological properties. Results presented in this paper have shown that Zr/Zr_2N coatings with an increased amount of ceramic phase have significantly better properties than Zr/Zr_2N coating with the same thicknesses of Zr and Zr_2N layers. The hardness of 8xZr/Zr_2N r1-4 coating H = 14.5 GPa is 30% higher than for 8xZr / Zr2N r1-1 coating. In spite of its higher stiffness, the greater value of the elasticity modulus, it exhibited greater scratch resistance. The critical load value is above 30N, while, for 8xZr/Zr2N r1-1, L_{C2} = 17.5 N. Moreover, both coatings with increased thicknesses of Zr_2N layers are characterized by a 2.5–3 times higher wear resistance. These results indicate that, in multilayer ceramics/metal coatings, ceramic layers should be thicker than metal ones. The 60 nm thickness of Zr layers is sufficient to effectively block the cracks formed in the ceramic layers. Too thick metal layers result in a lower hardness of the coatings and facilitate deformations due to their plasticity. A further process of testing of such systems is planned with coatings deposition with an even greater contribution of the ceramic phase, e.g., 1–8. However, as shown in literature studies, this may lead to the loss of the multilayer structure of coatings and the deterioration of their properties **[L. 4**, **8]**.

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