

PROPERTIES OF TOOL STEEL AFTER HYBRID TREATMENT CONNECTING DIFFUSION CHROMIZING WITH THE PVD METHOD

The objective of the research in this work was the modification of structure of carbide-type chromized layers, by the combination of diffusion chromizing with subsequent PVD treatment, consisting of chromium nitride deposition, carried out to improve their tribological properties. As a result, hybrid layers on the surface of tool steel were obtained. For comparison, the properties of single chromized carbide layers obtained in a diffusion chromizing process were tested. Investigations of layer microstructure, their mechanical properties, surface topography, adhesion of layers to the steel substrate, as well as tribological properties were conducted. The layer microstructure was characterized by X-ray diffraction and scanning electron microscopy. Topography of the layer surface was studied by an optical profilometer. The scratch test for investigations of layers adhesion to the steel substrate was used. Testing of tribological properties (linear wear) of the layers was performed by the three-cylinder-cone method. It was shown, that hybrid layers are characterized by a significantly smaller surface roughness than that of chromized carbide layers and their wear resistance improved almost twice with respect to carbide layers.

Keywords: Diffusion chromizing, PVD coating, carbide layer

1. Introduction

This paper concerns hybrid layers of the CrC+CrN type, produced on the steel surface in subsequent processes, diffusion chromizing combined with the next arc evaporation treatment Arc-PVD (Arc Physical Vapor Deposition), made for the deposition of chromium nitride coatings.

There exist numerous methods of diffusion chromizing, including powder pack, gas, salt bath, etc., [1-4]. Industrial significance in current times, has been gained by the inexpensive and technologically simple powder pack method (so-called pack cementation) which consists of heating the steel in a powder mixture containing ferro-chromium, which is a source of chromium, activator (e.g. ammonium chloride), and a ceramic filler (e.g. kaolin) preventing sintering of the powder at high temperature, [5-7].

The diffusion chromizing processes are carried out at temperatures within the range of 900 - 1100°C and times up to even 10 h, [1-5]. The chromized carbide layers (the CrC type carbide layer) are formed in the diffusion chromizing process on the surface of steels with a high carbon content. They are usually of small thickness, of the order of between 10 and 20 microns and are characterized by good tribological properties, [8-10].

In recent years, research has been undertaken to improve the properties of diffusion layers produced in thermo-chemical treatment processes, resulting from the growing requirements of

the industry regarding the durability and reliability of machine parts and tools, especially those working in difficult conditions. Meeting such requirements is possible through the modification of layer structure by the application of modern surface engineering techniques, [11-14].

The direction of investigations with a perspective is the combination of thermo-chemical treatment with PVD techniques, as well as other types of treatment, e.g. electroplating in processes of the duplex or hybrid type, [15-18]. An example here could be hybrid layers of the type nitrided layer/CrN coating with unique service properties, obtained in consecutive processes of gaseous nitriding, combined with a subsequent treatment: deposition of chromium nitride coating by arc evaporation (Arc-PVD) method, [19-21]. These layers characterized by, among other, high resistance to wear by friction, as well as high strength under heavy mechanical loading and high thermal shock resistance, have found application in the industry in the field of improving service life of forging dies made of hot work tool steel.

There are, however, not many publications on the subject of possibility of combining the diffusion chromizing process with subsequent Arc-PVD treatment, [22-23]. Investigations by the authors in this field have shown that from among various coatings deposited by the Arc-PVD method, characterized by good tribological properties, e.g. CrN, TiN, (Ti, Cr)N, Ti(C, N), best adhesion to the carbide surface of the chromized layer was exhibited by chromium nitride CrN coatings, [24].

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The aim of the research in this work was the modification of structure of carbide-type chromized layers, by the combination of diffusion chromizing with the next treatment arc evaporation (Arc-PVD), carried out to improve their tribological properties.

Investigations of the microstructure and tribological properties of the CrC+CrN type hybrid layers, produced as a result of this modification, as well as single carbide layers, were conducted. In addition, measurements of layer hardness, Young's modulus, surface topography and adhesion of layers to the steel substrate, were realized.

2. Experimental procedure

Chromized layers with a carbide microstructure (the CrC type carbide layers) were formed on samples of X210Cr12 tool steel (containing 1.90% C, 12.00% Cr).

The traditional powder pack method of diffusion chromizing has been described in detail in, [1,7]. The processes were carried out in an electric furnace, equipped with a temperature control system. Samples for the investigation were placed in a powder mixture containing: 60% ferro-chrome powder, 39% kaolin and 1% ammonium chloride (NH₄Cl), inside boxes made of heat resistant steel, of special design, with lids. The application of specially designed boxes enabled to achieve hermetic conditions during the process, with the aid of enamel which melts at temperature above 600°C, thus preventing from oxidation of the batch. Thus filled boxes were placed in the furnace, heated to temperature and soaked.

Following the process, where necessary, a thermal heating procedure was applied (austenitization at 990°C for 30 min, quenching and tempering at 200°C for 2 h).

Treatment of samples made from tool steel, indispensable for the formation of the CrC+CrN type hybrid layers, was carried out in two subsequent stages, utilizing two different equipment.

In the first stage, diffusion chromizing was conducted on the tool steel in order to form a CrC-type carbide chromizing layer on its surface. The diffusion chromizing processes were carried out in a GOAT-1200 electric furnace at the Institute of Precision Mechanics in Warsaw.

In the second stage, in order to form the CrC+CrN type hybrid layers, deposition was carried out of CrN chromium nitride on the surface of the chromized steel. The chromium nitride coatings were deposited by the arc-evaporation method (Arc-PVD), utilizing a Standard 1 type equipment at the Insti-

tute for Sustainable Technologies – NRI, in Radom. Process parameters of coating deposition by the Arc-PVD method are given in Table 1.

TABLE 1

Parameters of the chromium nitride deposition process

Type of process	Substrate temperature T [°C]	Substrate polarization voltage U _{BIAS} [V]	Pressure p [mbar]	Atmosphere
Etching by Cr ions	400	-950	5.0×10 ⁻³	Ar
Deposition of CrN	380	-150	3.5×10 ⁻²	N ₂

Two versions of heat treatment of the tool steel were applied in order to produce hybrid layers with different thickness.

In the first case, a hybrid layer of the CrC+CrN type with a total thickness of approx. 8 μm was formed, in which the thickness of the CrC carbide was approx. 7 μm, while that of the CrN coating – approx. 1 μm.

In the second case, a hybrid layer of the CrC+CrN type with a total thickness of approx. 16 μm was formed, in which the thickness of the CrC carbide was approx. 11 μm, while that of the CrN coating – approx. 5 μm.

Parameters of layers produced on the surface of tool steel are given in Table 2.

3. Methods of investigation

Investigations of layer microstructures was carried out on mounted and polished metallographic cross-sections. Microstructure of layers was evaluated with the aid of a Nikon LV150 optical microscope. The X-ray phase analysis was carried out with the aid of a Bruker D8 diffractometer operated at 40 kV and 40 mA with CuKα X-rays.

Scanning electron microscope studies, in conjunction with the analysis of chemical composition in micro-regions was carried out with the aid of a Hitachi TM-3000 scanning electron microscope, equipped with a BSE detector and an X-ray spectrometer with EDS. Hardness of the layers, as well as Young's modulus of the layers were measured by means of the Nano-Hardness Tester, of CSM Instruments, equipped with a Berkovich indenter.

TABLE 2

Parameters of layers formed on the surface of tool steel

No of version of hybrid treatment	Treatment stages	Stage I		Stage II		
	Type of process	Diffusion chromizing		Arc PVD treatment		
	Temperature /process time	Type of layer	Thickness of CrC carbide layer	Type of layer	Thickness of CrN coating	Thickness of CrC+CrN hybrid layer
1	900°C/4 h	CrC carbide	7 μm	CrC+CrN hybrid	1 μm	8 μm
2	900°C/10 h	CrC carbide	11 μm	CrC+CrN hybrid	5 μm	16 μm

Topography of the surface of samples was studied with the aid of the Bruker Contour GT-K1 optical profilometer.

For evaluation of adhesion of the layers to the steel substrate, the Revetest tester from CSM was chosen. This apparatus is equipped with a measuring head with a Rockwell indenter, an optical microscope with high resolution video imaging system, a sensor of acoustic emission, a penetration depth sensor, as well as a sensor of friction and normal forces.

Tribological properties of the samples, their wear resistance by friction, were evaluated by the three-cylinder-cone method, [25,26]. Measurements were taken with a rotating speed of the cone at $n = 576$ r.p.m. and unit loading pressures of 50, 100, 300 and 400 MPa during a time of 100 min, applying lubrication by Lux 10 oil.

4. Results and discussion

4a. Layer microstructure

X-ray diffraction phase analysis of the surface of chromized layers made from X210Cr12 tool steel revealed the presence of the $(\text{Cr, Fe})_7\text{C}_3$ type carbide, as well as traces of the $(\text{Cr, Fe})_2\text{N}$ nitride, similarly to what has been reported in other publications, [3,5,6].

X-ray diffraction phase analysis of the surface of samples with the CrC+CrN type hybrid layers, obtained by the deposition of the CrN coatings on the CrC carbide layer, revealed the presence of both the $(\text{Cr, Fe})_7\text{C}_3$ chromium carbide, as well as that of the CrN chromium nitride, Fig. 1. Similar results of X-ray analysis were obtained in the previous research realized by the authors, [17,23].

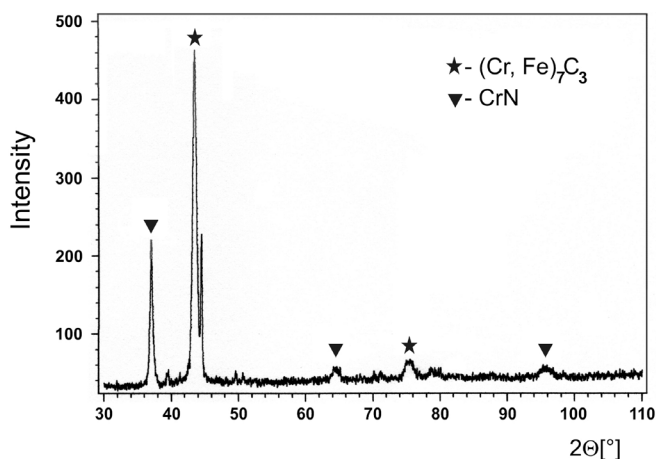


Fig. 1. X-ray diffraction pattern of the X210Cr12 steel surface with the CrC+CrN type hybrid layer

The surface of samples with hybrid and chromized carbide layers was silvery and smooth. Scanning image of the surface of steel sample with the CrC+CrN type hybrid layer, obtained with scanning electron microscope (Hitachi TM3000), is shown in Fig. 2. An analysis of the morphology of hybrid layer surface

showed the presence of a homogenous, very fine microstructure with a small amount of a droplet phase which is characteristic of the arc evaporation method, [21]. Similar scanning images of the surface of CrN coatings were also observed in other investigations, [27,28].

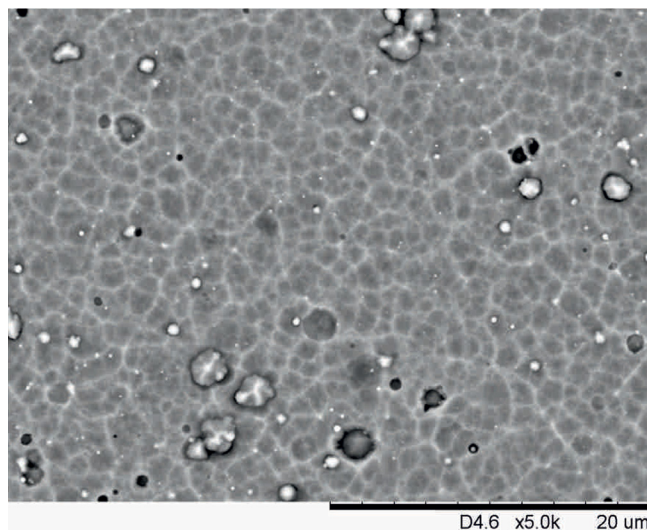


Fig. 2. The SEM image of the surface of the tool steel sample with the CrC+CrN hybrid layer

The scanning image (SEM+BSE+EDS) of tool steel sample with the CrC+CrN type hybrid layer, obtained on a metallographic cross-sections (Fig. 3), revealed the presence of two zones: the CrN chromium nitride coating, in the area adjacent to the surface of the layer and the chromium carbide in the area between the CrN nitride and the steel substrate. The chromium carbides (gray precipitations) are visible in the steel substrate, constituting the structural component of the X210Cr12 tool steel. An analysis of element concentration (chromium, iron, carbon and nitrogen) in selected micro-areas of this layer and its substrate, marked with points: 1 to 6 on Fig. 3, is shown in Table 3. The analysis of the chemical composition shows the presence of the CrN chromium nitride in the first zone (outer zone) of the hybrid layer, counting from the surface, because its main components are nitrogen and chromium (point 1 in Fig. 3 and Table 3). An enhanced concentration of chromium (approx. 64 wt.% Cr) relative to the concentration of nitrogen (approx. 34 wt.% N) may be caused by the presence of the droplet phase, containing chromium, which is formed at the surface of the CrN coating, obtained by the Arc PVD method, [27,29]. It should be emphasized that the result of the chemical composition analysis by the EDS method is the averaged result from the area where the electron beam of the scanning microscope works, which affects the error in determining the content of elements, in particular light elements, e.g. C or N. The maximum concentration of chromium in the zone of the layer between the CrN coating and the steel substrate, corresponding to the CrC chromium carbide, was approx. 60 wt.% Cr and decreased down to approx. 52 wt.% Cr with increasing distance from the surface (points 2-4 in Fig. 3 and Table 3). At the same time, the content of iron

increased from approx. 32 wt.% Fe to approx. 39 wt.% Fe. The carbon content remained constant at about 8 wt.% C, similar to the content of carbon in the type $(\text{Cr, Fe})_7\text{C}_3$ carbide, which is 9 wt.%, [1,5].

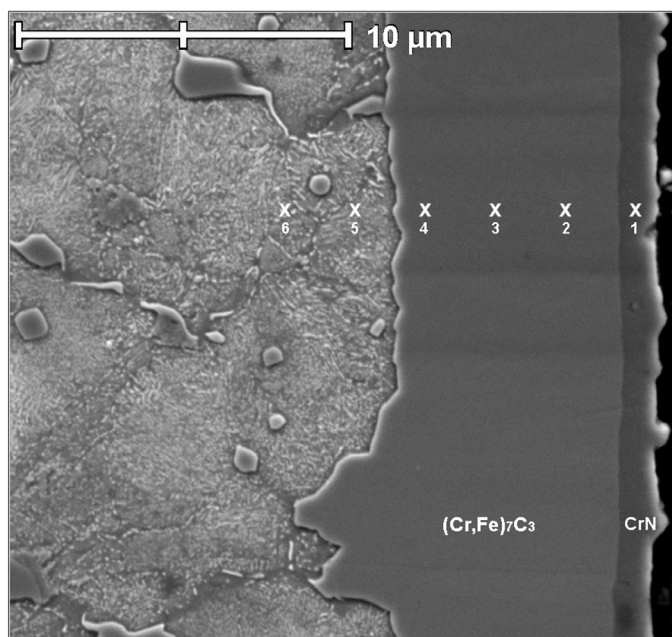


Fig. 3. Scanning image (SEM+BSE+EDS) of the CrC+CrN hybrid layer, produced on the surface of the tool steel, with micro-areas of the layer and its substrate, marked by points 1-6 on the image, analyzed by EDS (Table 3)

TABLE 3

Concentration of elements in micro-areas of the CrC+CrN hybrid layer and its substrate, marked by points 1-6 on the SEM image, Fig. 3

Chemical composition [weight %]				
Number of the point	C	N	Cr	Fe
1	0.50	34.31	64.46	0.73
2	8.41	0.00	60.01	31.58
3	8.58	0.00	56.50	34.92
4	8.32	0.00	52.32	39.36
5	2.04	0.00	8.55	89.41
6	1.98	0.00	9.72	88.30

Microscopic images of the CrC type carbide layer, produced on the surface of tool steel in the diffusion chromizing process (900°C/10 h; Table 2), as well as those of the CrC+CrN hybrid layer, obtained by deposition of chromium nitride coating on the surface of chromium carbide by the Arc PVD method, revealed by nital etching of metallographic cross-sections, are shown in Fig. 4a and Fig. 4b.

The bright carbide layer of the CrC type, with a thickness of approx. 11 μm, is separated from the substrate by a distinct boundary, Fig. 4a. In the substrate of the X210Cr12 tool steel, the etching revealed presence of chromium carbides, constituting structural components of that steel, which are well visible in phase contrast.

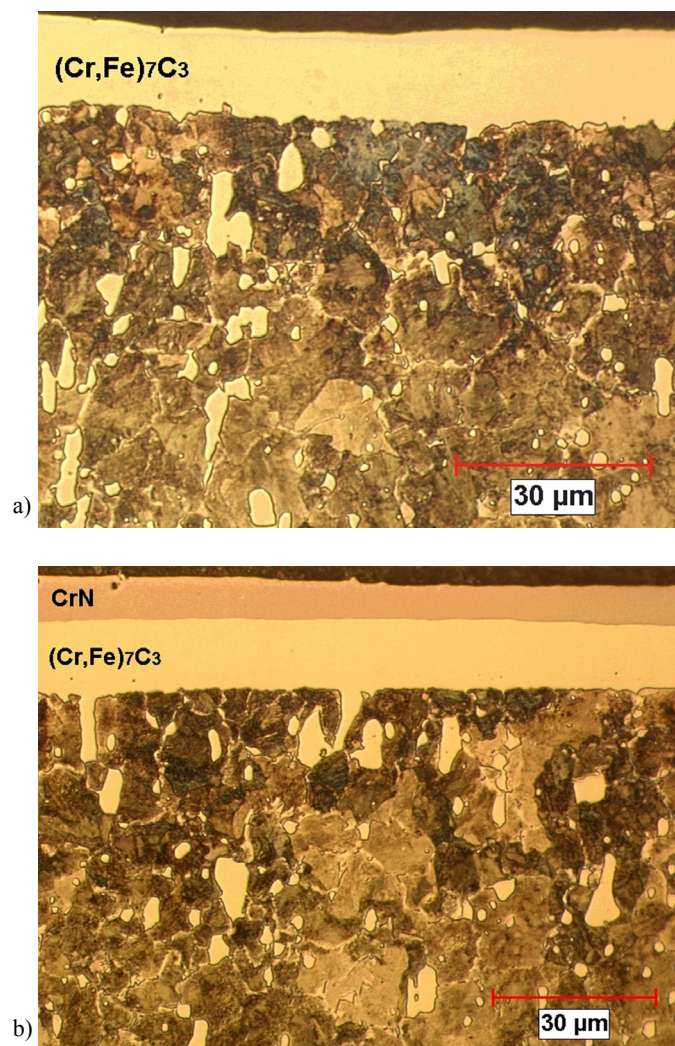


Fig. 4. Microstructure of the CrC type carbide layer (a) and the CrC+CrN type hybrid layer (b) formed on the surface of tool steel. Etched by 4% HNO_3

A microscopic image of the CrC+CrN type hybrid layer, with a thickness of approx. 16 μm, as already mentioned earlier, indicates its zone structure, Fig. 4b. The first zone (outer zone), counting from the surface of the sample, is the CrN chromium nitride, while the second zone (inner zone) is the $(\text{Cr, Fe})_7\text{C}_3$ chromium carbide, located in the area between the CrN zone and the tool steel substrate.

4b. Hardness, Young's modulus and surface topography

Measurements of hardness and of Young's modulus of samples prepared from tool steel, with hybrid layers of the CrC+CrN type, as well as those with only CrC chromium carbide layers, were carried out with the aid of a Nano-Hardness Tester, equipped with an indenter of the Berkovich type, maintaining maximum depth of indentation at less than 10% of the coating thickness. Results of measurements of hardness and Young's modulus, as well as of parameters which characterize surface

roughness, which were obtained with the aid of a profilometer, are all given in Table 4. Basic parameters which characterize surface roughness are S_a – arithmetical mean of absolute distances of the surface from the mean plane within the measured field, and S_z – the maximum height of roughness, i.e. average value of height of five highest peaks and five deepest valleys within the measured field, [30,31]. The roughness profiles are shown in Fig. 5.

TABLE 4

Results of measurements of hardness, Young's modulus and roughness

Type of layer	Thickness g [μm]	Hardness HV [Vickers]	Young's modulus E [GPa]	Roughness S_a/S_z [μm]
CrC+CrN hybrid	16 ± 1.4	2208 ± 55	329 ± 7	0.28 / 6.85
CrC carbide	11 ± 1.2	1846 ± 45	323 ± 6	0.52 / 10.58

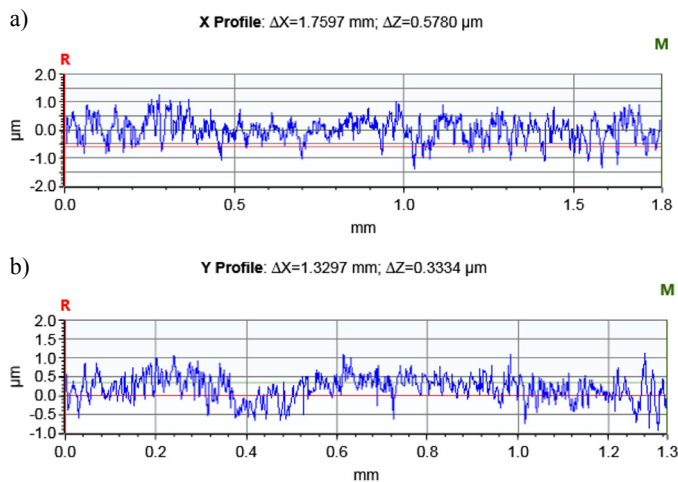


Fig. 5. Roughness profiles (a, b) of the surface of the CrC+CrN hybrid layer

An analysis of the material properties of samples with CrC+CrN hybrid layers, as well as CrC carbide layers, allows the conclusion that their Young's modulus, 329 GPa and 323 GPa, respectively, are similar within the limits of error, Table 4.

The hardness of the hybrid layer – 2208 HV is approx. 20% higher than that of the carbide layer, which is 1846 HV and that is most probably caused by the presence of the hard CrN phase on the surface.

In contrast, the surface roughness of the hybrid layer is almost half of that of the carbide layer, $S_a = 0.28 \mu\text{m}$ for hybrid layer and $S_a = 0.52 \mu\text{m}$ for carbide layer, respectively, Table 4.

4c. Adhesion

Investigation of adhesion of the layers was carried out by means of the scratch test, with gradual increase of the normal force loading the penetrator which scratch the surface of the

sample. The measure of adhesion was the critical force, i.e. lowest normal force causing the loss of adhesion of the coating to the substrate, [32].

Adhesion of layers was evaluated by the Revetest CSM tester, using load forces from 0 to 200 N, increasing at the rate of 100 N/min. The values of the force loading the penetrator at which destruction of adhesion occurs were determined based on a microscopic analysis of damages in the region of scratch, changes in the value of the coefficient of friction (μ) between the penetrator and the coating, as well as changes in the acoustic signal (AE) emitted in the process of scratch.

In the adhesion tests, the effect of penetrator loading on three different adhesion parameters was analyzed: Fc1, at which cracks are generated in the tested coating; Fc2, where local adhesion loss of the coating occurs in the area of the scratch and its edge, and Fc3, where decohesion of the entire layer from the substrate occurred. Three scratches were made on each sample.

Results of tests show good adhesion to the steel substrate of both the hybrid layers, for which the critical force $F_{c3} = 97 \text{ N}$, as well as for carbide layers, subject to destruction with a lower critical force $F_{c3} = 65 \text{ N}$, Fig. 6, Table 5.

TABLE 5

Critical load values determined during scratch test of the CrC type carbide layers and the CrC+CrN hybrid layers

Layer type	Critical load value, [N]			
	Scratch 1	Scratch 2	Scratch 3	Average
Carbide CrC	65	62	70	65
Hybrid CrC+CrN	106	98	86	97

4d. Tribological properties

Wear testing by the three cylinder-cone method, was carried out on samples of tool steel with the following types of layers:

- CrC-type carbide with approx. 11 μm thickness
- CrC+CrN – type hybrid with approx. 8 μm thickness
- CrC+CrN – type hybrid with approx. 16 μm thickness

For comparison, the wear resistance was also measured for hardened tool steel substrates without any layers.

The investigations showed that the linear wear of chromized samples, made of tool steel, with CrC carbide layers is twice smaller than that of steel samples, without any layers, subjected only to heat treatment, Fig. 7.

A subsequent treatment, the chromium nitride deposition by the Arc PVD method, carried out following diffusion chromizing, causes doubling of wear resistance by means of forming a hybrid layer. For example, linear wear of samples with the CrC+CrN hybrid layers (with a thickness of approx. $g=16 \mu\text{m}$) was 4.8 μm under 400 MPa units pressure for a friction time of 100 min, while that of the CrC carbide layer, under the same conditions was 10.5 μm.

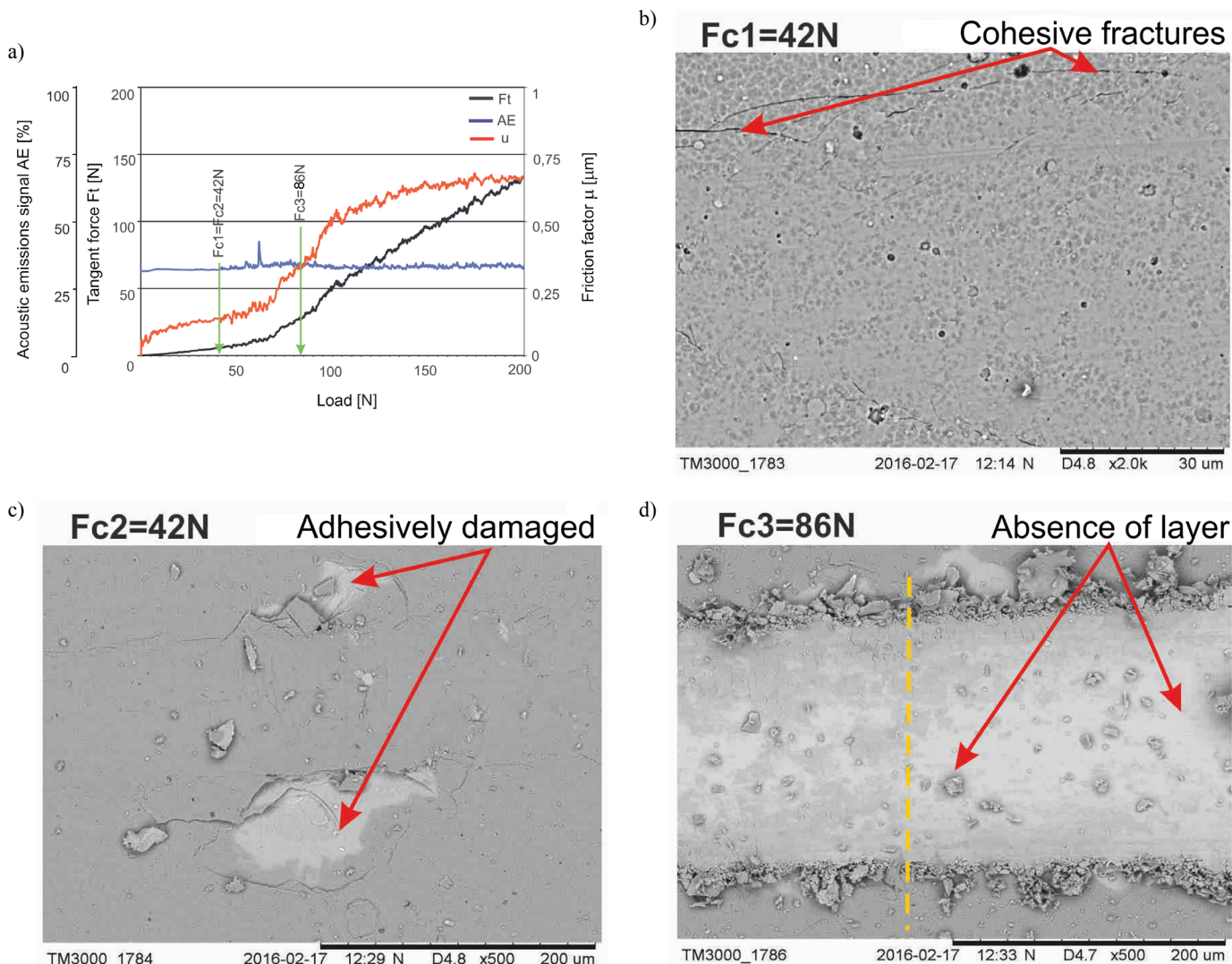


Fig. 6. Diagram of variations of the tangent force F_t , acoustic emissions signals AE and friction factor μ in function of load (a) and the SEM images of scratch (b, c, d), obtained during scratch test of the steel samples with the CrC+CrN hybrid layer

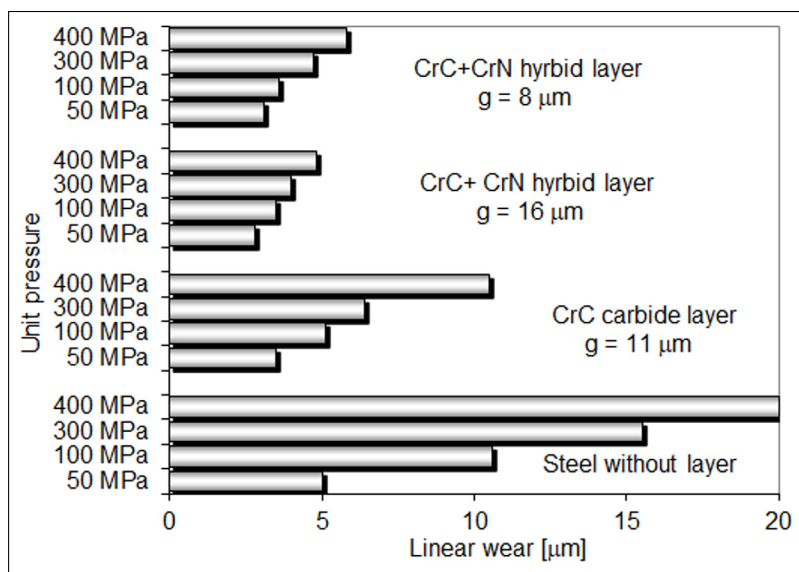


Fig. 7. Linear wear of the tool steel samples with the CrC+CrN type hybrid layers, the CrC type carbide layers and hardened steel samples without any layers, vs. units pressure

5. Conclusions

In the investigations the focus was on the modification of microstructure of carbide layers of the CrC type, accomplished by the combination of diffusion chromizing with a subsequent treatment consisting of CrN chromium nitride deposition by the Arc PVD method, in order to improve their tribological properties.

Diffusion chromizing of X210Cr12 tool steel by the traditional pack method enabled the formation of layers composed of the $(\text{Cr, Fe})_7\text{C}_3$ chromium carbide, containing trace amounts of the $(\text{Cr, Fe})_2\text{N}$ nitride, similarly to other known diffusion chromizing methods in which chromium halogens served as process activators, [5,8].

Thicknesses of the CrC carbide layers were approx. 7 and 11 μm, respectively for process times of 4 and 10 h, at a temperature of 900°C, Table 2.

The CrC+CrN hybrid layers, obtained by the deposition of a CrN coating by the Arc PVD method on the surface of the chromium carbide layer, consisted of two zones: the first (outer zone), containing the CrN nitride, and the second (inner zone), containing the $(Cr, Fe)_7C_3$ carbide, located in the area between the CrN coating and the tool steel substrate, Fig. 3, Fig. 4b. The thicknesses of the CrC+CrN hybrid layers were 8 μm (CrC – 7 μm ; CrN – 1 μm) and 16 μm (CrC – 11 μm ; CrN – 5 μm), Table 2.

Analyzing the material properties of the samples with hybrid and carbide layers, one can conclude that their Young modulus, 329 GPa and 323 GPa, respectively, are similar, Table 4. The hardness of the hybrid layer, 2208 HV is about 20% higher than that of the carbide layer which is 1846 HV. This is probably caused by the presence of the hard CrN phase at the surface, [21-23]. In contrast, the surface roughness of the hybrid layer, as evaluated by a profilometer, was found to be significantly smaller than that of the carbide layer, respectively $S_a = 0.28 \mu\text{m}$ for the CrC+CrN hybrid layer and $S_a = 0.52 \mu\text{m}$ for the CrC carbide layer, Table 4.

Similar values of Young's modulus, hardness and surface roughness of CrC carbide layers were reported in other publications, [7,33]. In both cases, the hybrid layers, as well as the carbide layers, showed good adhesion to the steel surfaces, as evaluated by the scratch test, Table 5, Fig. 6.

The investigations showed that the linear wear of tool steel samples with the CrC+CrN hybrid layers is two times lower than that of the CrC carbide layers, Fig. 7. This indicates the excellent tribological properties of the hybrid layers. The best tribological properties were exhibited by the CrC+CrN hybrid layer of approx. 16 μm thickness (accordingly: CrC – 11 μm ; CrN – 5 μm) the linear wear of which was 4.8 μm under unit loading force of 400 MPa, after 100 min of testing.

The results of the investigations showed a significant effect of surface roughness, expressed by the S_a parameter, on the frictional wear of tested layers. The CrC+CrN hybrid layers exhibited a surface roughness of almost two times the value of that obtained on the CrC carbide layers and, at the same time, exhibited better tribological properties, expressed by lower values of linear wear, as determined by the three cylinder-cone method. In other works, [28,29], similar relationships between tribological properties of layers and surface roughness were also found.

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