CHARACTERISTICS OF DIAMOND – LIKE CARBON(DLC) FILM DEPOSITED BY PACVD PROCESS

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Resume

Diamond – like carbon (DLC) film is promising materials for many technical and engineering applications. DLC films are used in many different industries for example: in medicine, in electronics, in optics and the automotive industry. They have excellent tribological properties (low friction coefficient), chemical inertness and high mechanical hardness.

This paper provides an analysis of the microstructure, mechanical and tribological properties of DLC films. In the study of the coating used several surface sensitive techniques and methods, i.e. High Resolution Transmission Electron Microscopy (HRTEM), Scanning Electron Microscopy (SEM), Raman spectroscopy and tribological tests like ball-on-disc.

HRTEM investigation shows an amorphous character of DLC layer. In sliding dry friction conditions the friction coefficient for the investigated elements is set in the range between 0.02-0.03. The investigated coating reveals high wear resistance. The coating demonstrated a good adhesion to the substrate.

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1. Introduction

Diamond-like carbon (DLC) is a meta-stable form of the amorphous carbon with predominance tetragonal bonds (sp3), characteristic of the diamond [1]. In practice, this term refers to a group of materials having both sp2 (graphite) and sp3 (diamond) bonds, but a number of sp3 bonds can vary from a few to tens of percent. The DLC coating includes both amorphous carbon (a-C) and hydrogenated carbon (a-C:H) [1, 2]. Depending on the ratio of sp3 to sp2 bonds and hydrogen content, DLC coatings are divided into several groups. The coatings of DLC type are: a-C, ta-C, a-C:Me, ta-C:H, a-C:H, a-C:H:Me (Me = W, Ti, Mo, etc.), a-C:H:X (X = Si, O_2, N_2) [3].

A major impact on the properties of DLC coatings is their way of deposition and humidity and the type of gas [4]. Various types of DLC coatings are different mechanical, electrical and tribological properties. Overall, DLC coatings have a high hardness, modulus of elasticity, thermal conductivity, optical transparency in the IR spectrum and electric resistance, abrasion resistance, good fracture toughness, chemical resistance, and biocompatibility [3-7].

The most important feature is the low friction coefficient (< 0.1) and wear resistance under the dry friction. Friction coefficient of DLC coatings also depends on the hydrogen content, the working environment and coating additives [8, 9]. This is related to the phenomenon of slip that occurs in the boundary layer through a process of graphitization and oxidation. The DLC coating is a kind of solid lubricant, consisted mainly of graphite and respective metalsoxides. During friction thanks to graphitization process the layer is renewable

[1]. However, as research showed graphitization is not the only factor causing the lubricating properties of DLC. The hydrogenated DLC has good lubricating properties under dry nitrogen and argon and vacuum, wherein the graphite has lost these properties [10].

It is important to control internal stresses in DLC coatings. DLC coatings have high compressive residual stresses (0.7 GPa – 5 GPa), which tends to result in plastic deformation and poor adhesion. Reducing the residual stress can be made by doping of metallic elements (Ti, Cu, W) or non – metal (O_2, N_2, Si). However, this causes a reduction of hardness. Compared to the monolayer, a multilayer coating maintains high useful properties while reducing the residual stress [10, 11]. Another way to reduce internal stress is surface texturing by segmentation of DLC films [12].

Because of the unique properties, deserves special attention use of low friction DLC coatings on coverage structural elements of machines and mechanical equipment. They are operating under variables tribological and corrosive conditions. The functionality of them depends largely on the structure and properties of surface layers. The coatings and thin layers are made for the protection of contact area and to counteract of material wear processes [13]. Conventional coatings used on tools are not suited to resist friction forces because they have not enough wear resistance [14].

The aim of the paper was to examine the structure, mechanical and tribological properties of DLC coatings deposited on the X40CrMoV5-1 hot work tool steel by plasma assisted chemical vapour deposition (PACVD) method.

2. Experimental procedures

The tests were made on samples of DLC (diamond-like carbon) coating deposited on the X40CrMoV5-1 hot work tool steel substrate. The DLC coating was produced by PACVD process. The DLC coating was deposited using acetylene (C2H2) as precursor. The deposition conditions were following: voltage: (-500 V), chamber pressure: 2 Pa, temperature process: 220ºC. To improve the adhesion of DLC coatings, a transition CrN interlayer was deposited.

Diffraction and thin film microstructure were tested with the use of the TITAN 80–300 ultrahigh resolution scanning/transmission electron microscope. The thin cross-section lamellas for transmission electron microscopy (TEM) observations were prepared by focused ion beam (FIB) technique using Quanta 200i instrument with gallium ions.

Adhesion of the coating to the substrate material was verified by the scratch test on the CSEM REVETEST device, by moving the diamond indenter along the examined specimen’s surface with gradually increasing load. The tests were made using the following parameters: load range: 0–100 N, load increase rate (dL/dt): 100 $N.min^{-1}$, indenter’s sliding speed (dx/dt): 10 $mm.min^{-1}$, acoustic emission detector’s sensitivity AE: 1.

The critical load L_{C2}, causing the loss of the coating adhesion to the material, was determined on the basis of the values of the acoustic emission *AE* and recorded friction force F_t as well as observations of the damage developed in the scratch test on a LEICA MEF4A light microscope.

The friction coefficient and wear rate of coating were determined in the ball-on-disc test. The tests were carried out on the T-01M (ITE) device with the following parameters: sliding speed: 0.2 $m{\cdot}s^{-1}$; normal load: 20 N; counterpart: Al_2O_3 of 10 mm diameter; sliding distance: 1000 m; temperature: 22°C (± 1°C); relative humidity: 30 % (± 5%).

3. Results and discussion

The cross-section of the investigated DLC coating is presented on Fig. 1. The coating presents a compact structure, without any visible delamination or defects. The morphology of the fracture of investigated coating is characterized by a dense microstructure. The fractographic observations made with the electron scanning microscope allow to state that the tested coating indicates a monolayer structure. The SEM investigation indicates the occurrence of CrN interlayer between the substrate material and the DLC coating, which affects the improved adhesion. The thickness of coating is 1.5 μm.

Subsequently, for the coating's structure characterization, the TEM and HRTEM observations were used. The images, presented on Fig. 2 were obtained from selected regions. The bright-field images (Fig. 2a) and HRTEM micrograph (Fig. 2b) show a nanocrystalline character of DLC film. However there are also areas present in the investigated layer of quasi-amorphous nature, what was confirmed by a mind of the electro diffraction investigation. The electron diffraction patterns obtained have shown the considerable broadening of diffraction rings (Fig. 2c).

The critical load values L_{C1} and L_{C2} were determined using the scratch test method with the linearly increasing load, characterizing adhesion of the investigated coatings to the steel substrate (Fig. 3). The first critical load L_{C1} corresponds to the point at which first damage is observed (Fig. 4a); the first appearance of microcracking, surface flaking outside or inside the track without any exposure of the substrate material - the first cohesion - related failure event. The second critical load L_{C2} is the point at which complete delamination of the coatings starts; the first appearance of cracking, chipping, spallation and delamination outside or inside the track with the exposure of the substrate material - the first adhesion related failure event (Fig. 4b). The investigated coating shows relatively high values of critical load. First failure occurs at values (~ 9 N). The second critical load values L_{C2} occurs at 37 N.

To determine the tribological properties of the DLC coating, an abrasion test under dry slide friction conditions was carried out by the ball-on-disc method. Fig. 5 presents the graph of friction coefficient μ changes obtained during wear tests in relation to counterpart with Al_2O_3. The friction curve has initial transitional state of unstabilized course, during which the friction coefficient is reduced along with the growth of sliding distance up to obtaining the stabilized state, which normally occurs after a distance of about 100 m. Under technically dry friction conditions, after the wearing-in period, the friction coefficient recorded for the associations tested is stabilized in the range 0.02-0.03. No case of complete coatings wear-through occurred because the maximum wear-in depths were below their thicknesses (Fig. 6).

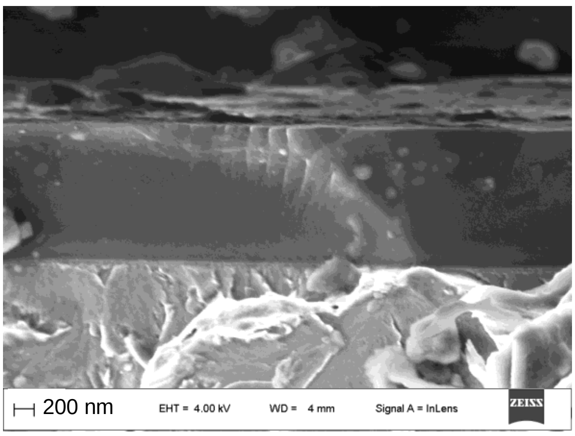
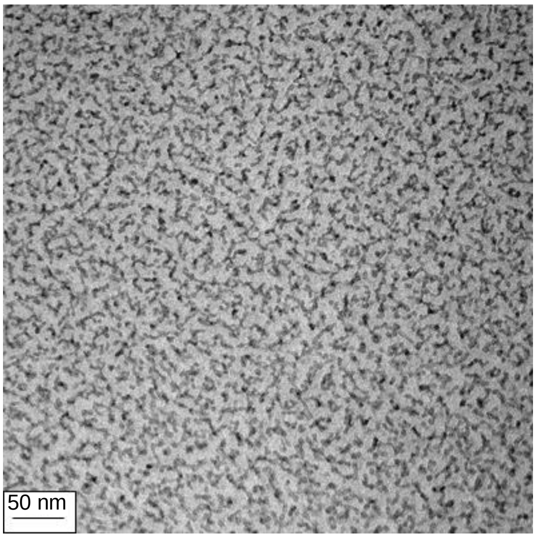
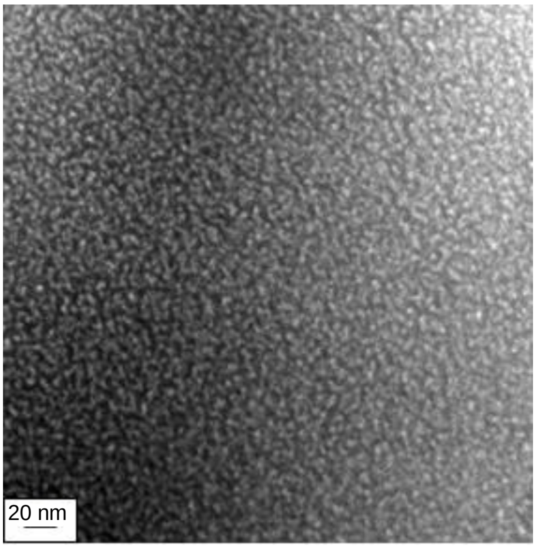


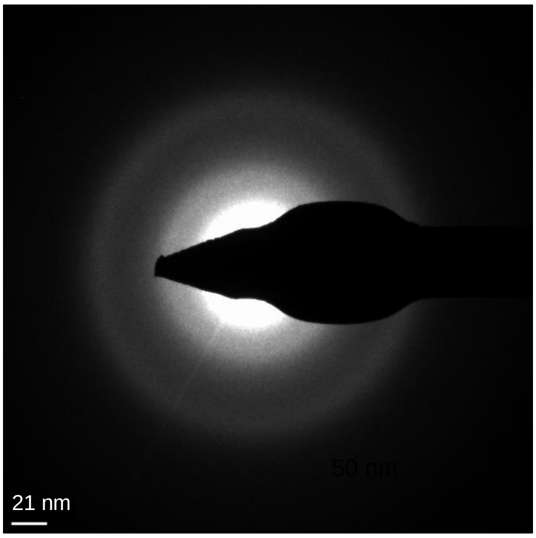
Fig. 1. SEM fracture image of DLC coating deposited onto the X40CrMoV5-1 steel substrate.



a) TEM bright-field image



b) TEM dark field image



c) corresponding SAED patter

Fig. 2. Structure of the DLC coating.

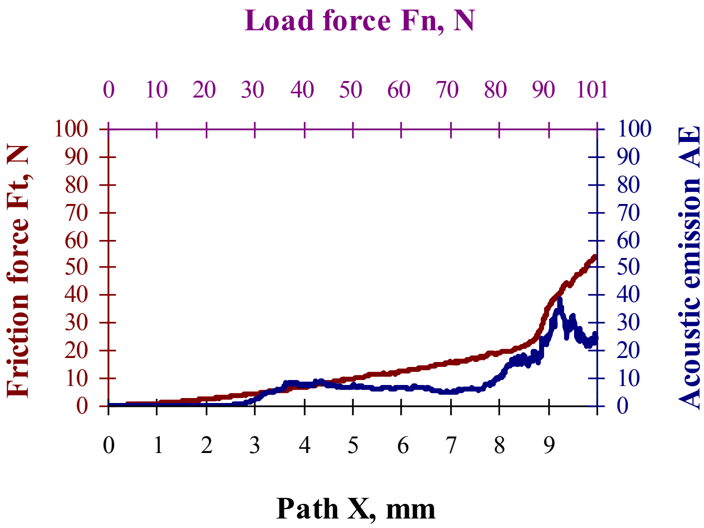
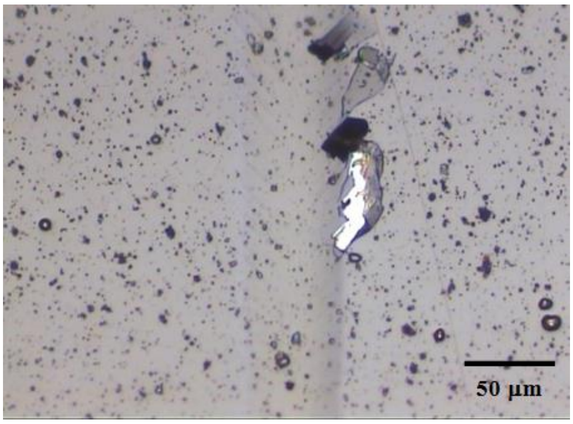
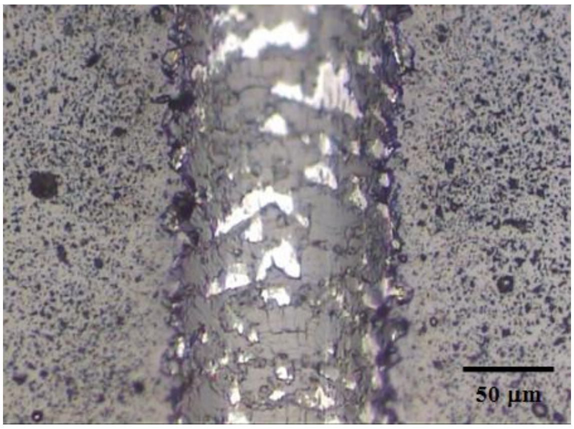


Fig. 3. Diagram of the dependence of the acoustic emission (AE) and friction force Ft on the load for the X40CrMoV5-1 steel with the DLC coating.

(full colour version available online)



a) at L_{C1}



b) at L_{C2}

Fig. 4. Scratch failure pictures of the DLC coating on X40CrMoV5-1 steel substrate.
(full colour version available online)

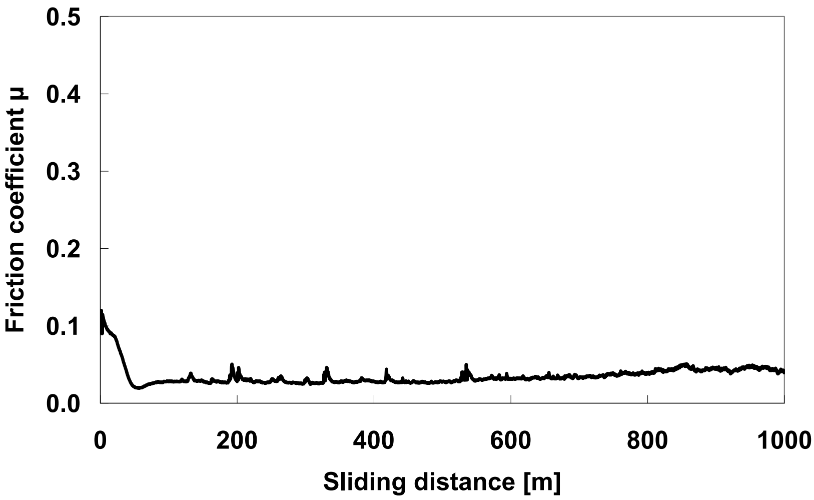


Fig. 5. Dependence of friction coefficient on sliding distance during the wear test for DLC coating.

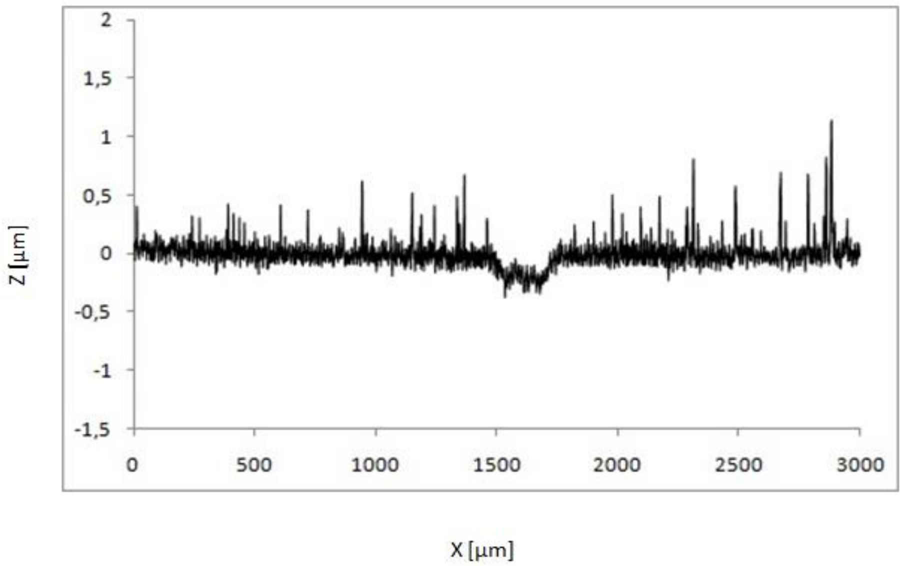


Fig. 6. Wear pattern profile after the wear test of DLC coating.

4. Conclusions

Basing on the investigation results the following conclusions were arrived at:

– DLC coating was deposited successfully on X40CrMoV5-1 hot work tool steel substrate;

– the TEM investigation revealed that DLC layers have a nanocrystalline and/or quasi-amorphous structure;

– on the basis of the scratch test, it was found that the critical load L_{C2} is in the range 37 N;

– under the technically dry friction conditions, the friction coefficient is within the range 0.02-0.03.

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