Microstructure and Tribological Properties of DLC Coatings

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Abstract. This paper presents the results of diamond-like carbon coatings deposited using the physical vapor deposition PVD process on 4H13 stainless steel samples. The properties were assessed by analyzing the coating microstructure, nanohardness, roughness and tribological tests. The results obtained during the tests which were carried out showed that the application of diamond coatings considerably improves tribological properties. In addition, coatings, which are desirable in sliding friction pairs, are free of pores and microcracks.

Introduction

Wear and abrasion are serious problems in all branches of industry. Although modern technologies permit considerable improvement of properties of the outer layer, they need to be modified or new solutions have to be looked for [1, 2].

Functional properties of many elements of machine parts depend not only on the possibility of transferring mechanical loads through the entire cross-section of the material, but mainly on the structure and properties of surface layers. The increasing demands placed on construction materials have led to constant attempts to produce new types or modifications of wear protection, including changing the chemical composition of a coating or the technology of its production [3-5]. To properly protect surfaces and reduce wear processes, top layers and coatings must perform the following functions [6]:

- reduce consumption,
- prevent and reduce direct contact of metal components,
- facilitate tangential movement by reducing friction forces,
- cause the distribution of normal forces over the largest possible nominal contact area,
- suppress vibrations and oscillatory movements.

Applying protective coatings to machine parts is economically justifiable if the wear is local or if the coating material is expected to display properties different from those of the substrate. Most surface layers are technological surface layers (TSLs) - they are produced before objects are used. Functional surface layers (FSLs), on the other hand, are applied during maintenance.

In recent years there has been great progress in the development of research and application topics related to carbon materials. It includes obtaining diamond-like coatings, DLC (Diamond Like Carbon) and applying them by PVD and CVD methods [7-9]. Carbon creates the most chemical compounds among all elements and has several allotropic varieties.

Due to their special properties, diamond-like carbon coatings are used, among others in sheet metal pressing, steel forming, aluminum foundries, in the production and regeneration of tools and devices, in the production of machine parts and components for the automotive industry. They are also used in the rubber and food industry.

The work discusses the properties of diamond-like carbon coatings deposited using the PVD process. The properties were established based on the results of a microstructure analysis, nanohardness and roughness tests and tribological studies.

There are many alternative technologies for producing coatings and improvements of material properties in relation to PVD technology [10]. The analysis of properties of DLC coating systems requires many methods [11, 12].

Due to its unique and characteristic properties, DLC coatings can be used wherever hardness, wear resistance, slipperiness are required, so primarily hydraulic parts [13], including those heavily mechanically [14-16] and thermally [17-19], as well as exposed to cavitation erosion [20]. The introduction of DLC coatings favorably changes the operational features of machine parts, and in particular reduces the number of failures and non-conformities, which has a great impact on the planning and optimization of production in many industries, such as power plants [21], production of industrial and consumer films [22] and parts of industrial robots [23]. At the same time, these atypical coatings influence the development of research methods in both experimental [24] and data analysis [25].

Experimental

Materials with anti-wear functions intended for work in friction nodes covered with diamond-like carbon (DLC) coatings type a-C:H with W and Cr interlayer obtained in physical vapor deposition processes, PVD, were selected for research. The choice of DLC coatings was dictated by their excellent properties and the possibilities of a very wide application in various industries. Amorphous hydrogenated carbon (a-C:H) film, also known as diamond-like carbon coating, is characterized by excellent mechanical properties.

The substrate material was used of 4H13 stainless steel. The elemental composition of the steel used was as follows (wt.%): C: 0.36-0.45, Mn: 0.50-0.80, Cr: 12.0-14.0, Si: 0.60-0.80, Mo: 0.5-0.7, V: 0.2-0.3, Ni: 0.1-0.60, P: max 0.04, S: max 0.03, and the rest is iron.

The processes of applying thin PVD anti-wear coatings take place at elevated temperatures, which causes tempering of the surface layers and reduction of hardness. Individual coatings were obtained in the following processes and temperatures:

- a-C:H in the physical vapor deposition process PVD by ion spray at a temperature < 300° C,
- substrate material temperature of 350° C.

Results and discussion

A microstructure analysis was conducted for DLC coating using a *JEOL* JSM-*7100F* scanning electron microscope with field emission. Figure 1 shows the microstructure of DLC coatings. The layer thickness is approximately 1.1 μm. In the photograph, the boundary line between the coating and the substrate is clear. The coating is free of pores and microcracks.

The element maps of an amorphous hydrogenated carbon film on the examined stainless steel surface are shown in Fig. 2. It turned out that the element maps of Fe, Cr, W and C are clearly visible on diamond-like carbon films. Furthermore, all the element distributions are heterogeneous and some of the grains are oxygen deficient. Moreover, the DLC film covers a significant portion of the specimen surface.

Production of coatings with the required micromechanical properties is a major research challenge. In the production process, it is necessary to specify the controlled parameters and properties that we expect from the resulting coating.

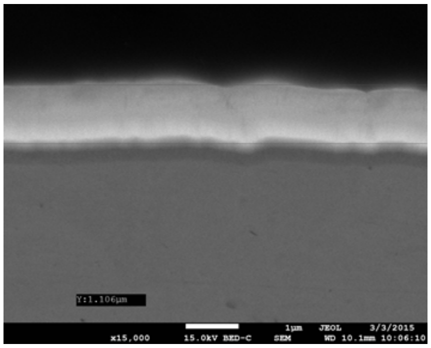

Fig. 1. Microstructure in the DLC coating

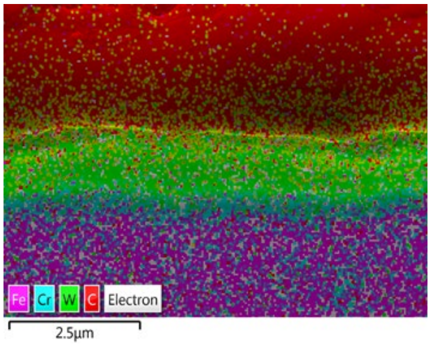

Fig. 2. Maps of elements in DLC coating

The multitude of controlled parameters and their values mean that a large number of combinations results in high costs. In our case, nanohardness and elastic modulus (Young's modulus) of DLC coatings were tested. The hardness and elastic modulus were investigated by nanoitender technique. This Measurement technology was possible due to the development of instruments that continuously measure force and displacement. The tests were carried out with the following parameters: linear load, max. load of 3.2 mN, load and unload speed of 40 mN/min as well as a break time between successive cycles of load and unload of 3 s. On the basis of 10 measurements, the values of average nanohardness and elasticity modulus were determined and placed in Table 1. This table contains the average values of nanohardness and elastic modulus together with the standard error.

Table 1. Value of nonohardness and modulus of elasticity with errors

Material	Nanohardness [GPa]	Elastic modulus [GPa]
DLC coating	7.55 ± 0.10	92.87 ± 2.05
4H13 steel	5.60 ± 0.40	44.00 ± 4.30

While analyzing Table 1, it can be concluded that the DLC coating nanohardness was approx. 26% higher compared to the 4H13 stainless steel nanohardness. A similar analogy can be observed by analyzing the values of Young's modules for DLC coating and 4H13 stainless steel.

Investigations into dry friction resistances were performed using the T-01M pin-on-disk type tribotester. In Fig. 3, the T-01M principle of operation is shown. The specimens were rings of 4H13 stainless steel, onto which DLC coatings were PVD method deposited. The counter specimen was a ball, ϕ6.3 mm in diameter, made of 100Cr6 steel. Tribological tests were conducted using the following parameters:

- linear speed, $v = 0.8$ m/s,
- test duration, $t = 3600$ s,
- range of load changes, $Q = 4.9; 9.8; 14.7$ N.

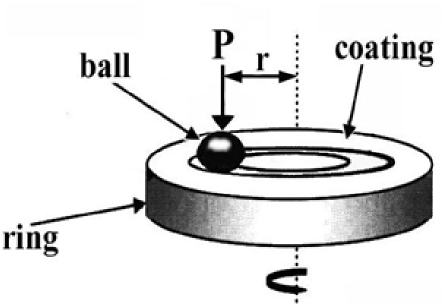

Fig. 3. Operation of the pin on disc type tester

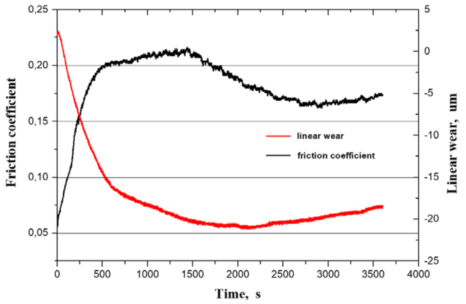

Fig. 4. Friction coefficient and linear wear as a function of time

An exemplary graph (Fig. 4) shows profiles of friction coefficient and linear wear as a function of time for the load of 4.9 N. The graph presented in Figure 4 refers to the tests on DLC coating. In dry friction, in the examined coating, the technological surface layer (TSL) was transformed into a functional surface layer (FSL). The effect was produced mainly due to sliding stresses and speed and the action of the atmosphere of the environment close to the tested surface. The stabilization of the state of anti-wear surface layer was observed (AWSL). In the profile (Fig. 4) that refers to the DLC coating, it can be seen that the stabilization of the friction coefficient takes place after approx. 2700 seconds, the stabilization value ranges between 0.16-0.18. The course of linear wear is exponential.

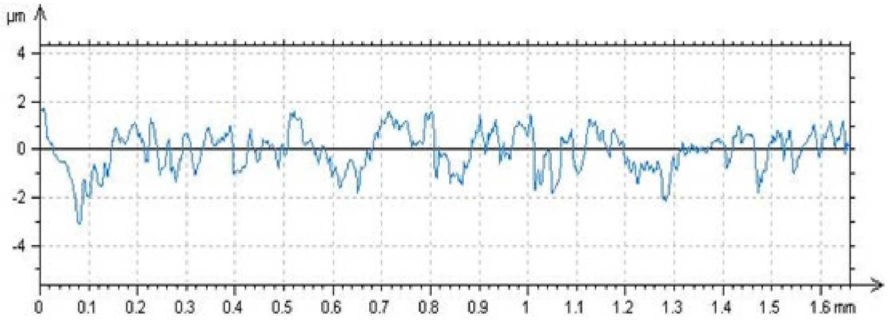

Fig. 5. View of roughness profile of DLC coating

Table 2. Results of roughness profile of DLC coating

Parameters of roughness	DLC coating	Parameters of roughness	DLC coating
Rp [µm]	1.81	Ra [µm]	0.66
Rv [µm]	2.33	Rq [µm]	0.81
Rz [µm]	4.14	Rsk	-0.39
Rc [µm]	1.91	Rku	2.74
Rt [µm]	4.86	–	–

The roughness of the DLC coatings was measured at the Laboratory for Measurement of Geometric Quantities of the Kielce University of Technology using a TALYSURF CCI equipment. The roughness was measured in two directions perpendicular to each other. Then, the average value Ra = 0.60÷0.66 µm was calculated. The steel specimens (4H13) without coatings had a roughness from 0.41 to 0.43 µm. Fig.5 presents an example two-dimensional surface microgeometry measurement of the DLC coating. Table 2 presents the most important average roughness parameters of the tested coating system. Low value of roughness parameters of DLC coating also has influence on mechanical properties.

Summary

DLC coatings are characterized by good mechanical properties. They have a homogeneous structure and are free of defects. The layer thickness was approximately 1.1 µm. The average value of the friction coefficient (at the moment of stabilization) obtained during the tribological tests for a DLC coating were between 0.16-0.18. DLC coatings are characterized by low roughness and high nanohardness. Further research will be targeted at the determination of corrosion and erosion resistance.

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