Adhesion Properties Evaluation of Heat Treated Steel 100Cr6 with Applied DLC Coating

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Abstract. The article deals with determination of tribological and basic properties of heat-treated bearing steel 100Cr6. It is typical steel for production a small and medium size of rolling bearings. For reducing the friction coefficient of functional surfaces of heat-treated bearing steel 100Cr6, the application of DLC (Diamond Like Carbon) coating is used. The quality of the thin surface coating was evaluate in terms of adhesion-cohesive properties. The measurement of the mechanical properties nanohardness, modulus of elasticity was perform by the nanoindentation method. A scratch test was use for analysis of adhesion properties. All results of experimental measurements will suitable for design of carry materials and iron-based materials. The work was done by financial support of project No. KEGA 026ŽU-4/2019 Implementation of integrated GPS system for specification and products verification into the teaching process of engineering study programs and putting them into the technical practice at the University of Žilina, Department of Mechanical Design and Machine Elements.

Keywords: Heat Treated 100Cr6 · Adhesion · DLC Coating · Nanoindentation · Scratch Test

1 Introduction

At present, reducing the wear of functional parts surfaces is also achieved by methods and procedures aimed at improving the mechanical and tribological properties of the surfaces themselves. One of the most widespread methods is the application of special types of thin coatings and coatings that are applied to the surface of a heat-treated substrate. The focus of development and preparation of PVD (Physical Vapor Deposition) coatings in recent years has changed in terms of their application as quickly as the demands for new coatings are changing. For this reason, traditional PVD coatings are optimize in a progressive manner. In terms of the principles of technologies as PVD, DCMS (Direct Current Magnetron Sputtering), HiPIMS (High Power Impulse Magnetron Sputtering) the range of materials and the structure of PVD coatings, the changes made more sophisticated and are not jumping in the short term. This means that changes have a life cycle character and the structures used and their material composition are significantly timeless [1–4].

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In actual industrial practice, combinations of very hard layers with sliding layers are developed, which are also functional surfaces. In addition, the technology of mechanical treatment of substrates before and after coating, which significantly contributes to improving the performance of the coated substrates, is also being developed and applied. [2]. These methods and procedures make significant progress in increasing of durability and lifetime of tools produced from, for example, high speed steels and sintered carbides [1–3].

An important factor in coating formation is the adhesion of the coating to the substrate material. In order for the coating to function properly, it must adhere perfectly to the substrate. The composition of the coating layer expects to not only higher microhardness values and other mechanical properties, but also higher thermal and chemical resistance. Its application to tribological properties are expected to be characterized by a low coefficient of friction against steel, low application temperatures on the substrate and removal of lubricating fluids in the systems [2, 5].

For the measuring of adhesion properties (strength of connection between coating and substrate) was used an experiments as scratch test and nanoindetation [1, 6, 7].

2 Materials and Methods

The experimental material is standard hardened bearing steel 100Cr6. The chemical composition of used bearing steel is present in Table 1. Chemical composition is in accordance with EN ISO 683–17:2014 [8].

The samples of 100Cr6 bearing steel were heat treated with the recommended mode, which is characteristic for this steel [5].

2.1 Heat Treatment Condition

The temperature of Austenitization was 830 °C with holding time 25 min. Subsequently, the samples were quench by oil DURIXOL V71ed®. The tempering of the samples was perform at 160 °C for 2 h. Heat treatment of the samples resulted in a structure consisting of tempered Martensite with uniformly distributed carbides $(Fe, Cr)_3C$ and residual austenite.

2.2 Microstructure Analysis

The microstructure analysis of heat-treated samples was performed by standard metallographic methods. As final samples preparation for analysis microstructure, samples were

Table 1. Chemical composition of steel 100Cr6

Chemical composition [hm. %]							
C	Si	Mn	P	S	Cr	Ni	Fe
1.05	0.35	0.45	0.0029	0.0033	1.55	0.182	-

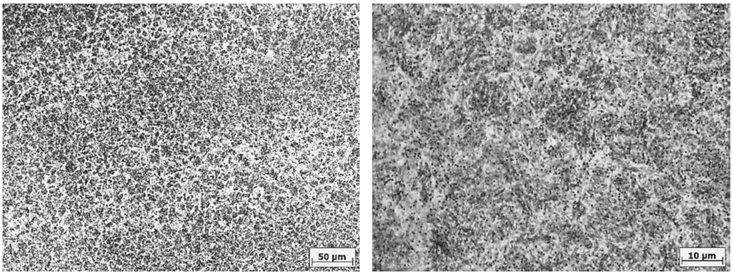

Fig. 1. Microstructure of heat-treated 100Cr6 bearing steel after heat-treatment, etched by 0.5% Nital.

etched by 0.5% Nital. Characteristic microstructure and detail of heat-treated bearing steel 100Cr6 is presented (see Fig. 1).

Hardness measurement. The hardness measurement was evaluated according to the Rockwell method, after heat-treatment. The average hardness value was measured 63 HRC.

DLC coating deposition. The heat-treated bearing steel 100Cr6 tends as a substrate material for deposition of DLC coatings. The coating deposition process was carried out at 240 °C for 6 h in a vacuum chamber where the working pressure range was from 0.1 to 1 Pa. Due to the coating at temperature 240 °C, the hardness value decreased to 59 HRC.

The aim of the experiments was to increase the performance of heat-treated bearing steel 100Cr6 by suitable treatment of its surface [6, 7, 9]. With PVD coating technology and magnetron sputtering, a Carbon-X coating (type DLC coating) was deposited on the surface of heat-treated bearing steel 100Cr6 substrate. The purpose of the experiment was to determine the adhesion and mechanical properties of the Carbon-X coating. The expected results were improvements of the tribological properties of heat-treated bearing steel 100Cr6 and an increasing of wear resistance of the surface.

2.3 Scratch Test

After applying of thin coating on the substrate, the scratch tests performed to evaluate the adhesion of the coating to the substrate. Scratch tests performed on a Bruker UMT® with a Rockwell standard diamond tip at a maximum load of 60 N and on a track with length 6 mm. The scratch tester connected to the computer via a control device. The adhesion-cohesive properties evaluated from the morphology of the scratch test and the graphical recording of the acoustic emission signal AE, the coefficient of friction COF depending on the load value and the optical image [10]. Three tests performed on one sample to minimize the effect of local defects that could affect the results of the adhesive layer assessment.

2.4 Nanoindentation

The measurements of the mechanical properties were carried out on heat-treated bearing steel 100Cr6 and Carbon-X coating. The nanoindentation measurements were performed by Nanoindentation Tester Anton Paar NHT®, with a standard Berkovich® diamond tip. The H_{IT} hardness and E_{IT} elastic modulus were measured and evaluated. The indentation matrix was 4x3, where 12 measurements were made on the sample. The maximum load for each measurement was decided so that the maximum penetration depth in the coating did not exceed 10% of its supposed thickness value [11, 12]

3 Results and Discussion

3.1 Adhesion of Coating Carbon-X Measurement Results

The resultant L_{C1} and L_{C2} adhesion values of Carbon-X are present in Table 2, wherein L_{C1} is the first cracking load and L_{C2} is the delamination load [13]. The adhesion-cohesive properties of the coating system were evaluated in combination with the acoustic emission patterns; friction coefficients and optical image (see Fig. 2, Fig. 3 and Fig. 4).

The Graph (see Fig. 2) shows the acoustic emission record AE depending on the normal load F_Z applied from the indentor.

The significant increase in the acoustic emission signal was determined by the adhesion-resistant failure of the Carbon-X coating. The acoustic emission values found in the measurement No. 001 was not included in the evaluation. It was probably a measurement artefact because the signal was not caused by cracks. From the measurement No. 002 revealed to the normal force load FZ ~ 26 N a significant value of the acoustic emission signal was evaluated. From the evaluation of the sample record No. 003, FZ ~ 41 N was found. The calculated mean value of the occurrence of the first cracks was FZ = 33 N.

According to author [11, 13], a protection of elements under mechanical stress is satisfactory for coatings used in operation condition with values of adhesion force FU $\leq$ 45 N.

The Fig. 3 presents the graphical dependence of the coefficient of friction COF vs. the load force magnitude FZ. The track surface morphology after scratch tests presents Fig. 4.

The assessment of the results in Fig. 3 and Fig. 4 shows that a significant increase in the coefficient of friction is associated with the delamination of the coating and the indentor tip penetrated the coating into the substrate.

For sample No. 001, the friction coefficient began to increase at a loading force of FZ ~ 42 N, for sample No. 002 at FZ ~ 30 N, and for sample No. 003 at FZ ~ 41 N. The average critical force FZ required to break the Carbon-X coating was determined as FZ = 38 N.

Table 2. Measurement results for adhesion of coating Caron-X

Adhesion	No. 001	No. 002	No. 003	Average value
L_{C1}	-	26	40	33
L_{C2}	44	30	41	38

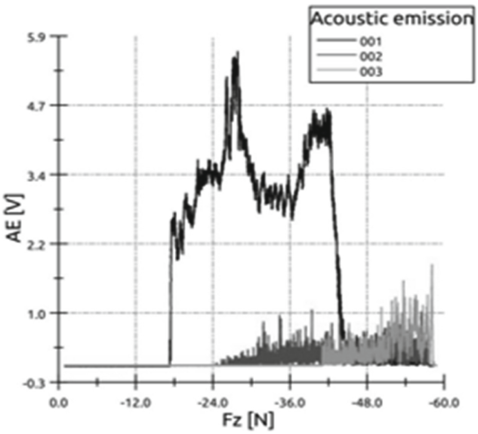

Fig. 2. Scratch test, graphical dependence of acoustic emission AE vs loading force FZ for coating Caron-X.

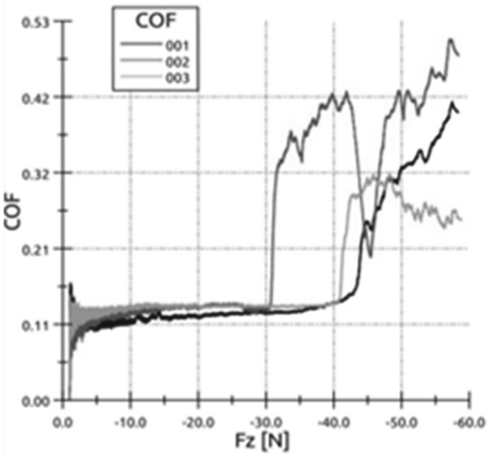

Fig. 3. Scratch test, graphical dependence of friction coefficient COF and loading force FZ for coating Caron-X.

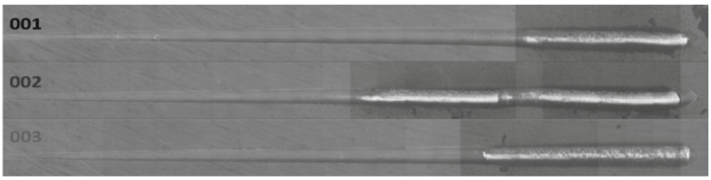

Fig. 4. Morphology of tracks after scratch tests on sample with coating Carbon-X, for measurements No. 001, No. 002, No. 003.

Table 3. Measurement results of mechanical properties of heat-treated 100Cr6 bearing steel and coating Carbon-X, [GPa]

Material	Steel 100Cr6		Carbon-X coating	
Measurement	H_{IT}	E_{IT}	H_{IT}	E_{IT}
1	9.83	235.6	7.36	106.5
2	9.72	238.9	9.31	132.5
3	10.1	249.4	8.12	107.2
4	9.87	233.3	9.3	130
5	9.87	247	7.76	104.4
6	9.9	243	7.99	107.2
7	10.03	253.6	9.64	123
8	10.61	235.4	8.55	106.7
9	9.51	251.5	8.25	104.7
Average value	9.9 ± 0.3	243 ± 7	8.5 ± 0.7	112 ± 11

3.2 Nanoindentation Measurement Results

The results obtained by measuring the mechanical properties of heat-treated 100Cr6 bearing steel and Carbon-X coating using the nanoindentation method are given in Table 3. There are presented the average values of the selection for hardness HIT with the appropriate standard deviation and for modulus of elasticity EIT with the appropriate standard deviation.

The graphical dependences of measured mechanical properties of heat treated 100Cr6 bearing steel and the Carbon-X coating by the nanoindentation method is presented in Fig. 5 and Fig. 6. 100Cr6

For heat-treated 100Cr6 bearing steel, the maximum penetration depth was approximately the same as for Carbon-X coatings. Gradual loading up to the maximum value of normal force took time 20 s; a maximum load, normal force value was token by 5 s. Then followed unload process took time 20 s.

Load curves were evaluated by the method developed with Oliver and Pharr. The method we introduced in 1992 for measuring hardness and elastic modulus by instrumented indentation techniques and has widely been adopted and used in the characterization of mechanical behavior of materials at small scales [14]. For all measurements, the Poisson ratio $\nu = 0.3$ was used.

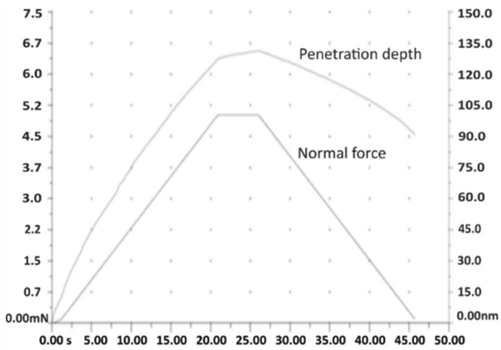

Fig. 5. Nanoindentation, graphical dependence of time vs. normal force and penetration depth for of heat-treated 100Cr6 bearing steel.

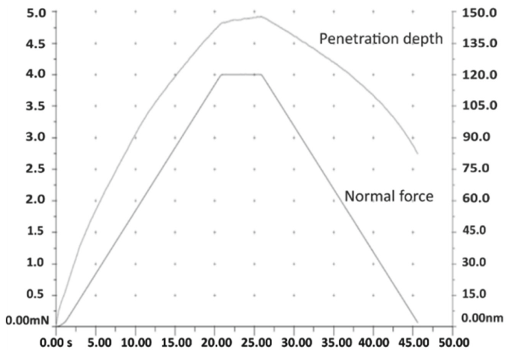

Fig. 6. Nanoindentation, graphical dependence of time vs. normal force and penetration depth for heat-treated for Carbon-X coating.

4 Conclusions

The aim of further experiments will be to verify the frictional properties of the Carbon-X coating. The quality substrate and the perfect final preparation of its surface before the application of the thin coating guarantee good performance properties of the coated components in terms of their mechanical stress under operating conditions.

Carbon-X coating has lower nanohardness and modulus of elasticity as heat-treated 100Cr6 bearing steel. Nevertheless, it has good adhesion-cohesive properties to the heat-treated 100Cr6 bearing steel substrate. The average value of force at which the first cracks occur in the Carbon-X coating is FZ = 33 N and the average value of critical force FZ required to break the Carbon-X coating is FZ = 38 N. The average value of heat-treated 100Cr6 bearing steel nanohardness is HIT = 9.9 ± 0.3 GPa and the modulus of elasticity EIT = 243 ± 7 GPa. The average Carbon-X hardness value HIT = 8.5 ± 0.7 GPa and the modulus of elasticity is EIT = 112 ± 11 GPa.

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