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Influence of laser texturing on tribological properties of DLC coatings

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Abstract
The work presents the use of laser texturing of DLC coatings to improve tribological properties. The coatings were applied by the PVD method to the rings made of 4H13 steel. The surface texturing was performed with the TruMICRO 5325c picosecond laser with the radiation wavelength λ = 343 nm. The surface microstructure analysis, surface microgeometry and microhardness measurements and tribological tests were carried out. The problem presented in the paper can be used to extend the knowledge of the areas of application of DLC coatings, especially in sliding friction pairs.

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1. Introduction

Diamond like carbon (DLC) coatings are a metastable form of amorphous carbon that contains sp^3, sp^2 and sp^1 bonds. DLC coatings were first developed in 1970 by Aisenberg and Chabot. The new structures were obtained by chemical and physical deposition methods with the addition of an ion beam to improve the coating properties (Ozimina et al., 2012).

An important factor on which the properties of the coatings depend is the ratio of sp^3 to sp^2 bonds. The sp^2 hybridization is responsible for good electrical conductivity and low coefficient of friction, while the sp3 hybridization determines high hardness, chemical inertness, and high wear resistance.

DLC coatings can be classified with regards to the amount of sp^3 bonds and hydrogen content as (Madej, 2013):

- ta-C (tetrahedral amorphous carbon)- maximum sp^3 bond content, no hydrogen present, has a stable and smooth form, and is the hardest of all coatings,
- ta-C:H- has about 70% sp^3 bonds, hydrogen content 25-35%,
- a-C- has up to 30% sp^3 bonds, no hydrogen present,
- a-C:H- has 20-40% hydrogen.

Furthermore, a-C coatings can be modified with metals (a-C:Me a-C:Me coatings), and a-C:H coatings both with metals (a-C:H:Me coatings modified with Ti, Nb, W, V, Cr, Co, Mo) and nonmetals (a-C:H:X coatings modified with Si, F, H, N, O, B, P).

Figure 1 shows the types of amorphous carbon depending on the hydrogen concentration and the proportion of covalent bonds.

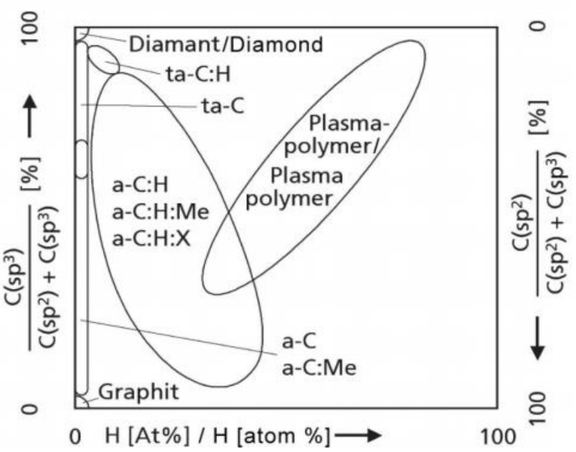

Fig. 1. Types of amorphous carbon as a function of hydrogen content and sp^3 and sp^2 bonds (Madej, 2013)

Due to their unique properties such as high hardness, low coefficient of friction, chemical inertness, abrasion resistance as well as biocompatibility, i.e. no negative impact on the human body, DLC coatings are very popular (Madej, 2014; Sanchez, 2000; Mansano, 2010). DLC coatings are used to cover tools for cutting and grinding, plastic processing as well as implants and surgical instruments (Esteve, 1999). DLC films have been also developed for many other fields. Their unique properties enable increased corrosion protection (Lipiński, 2017; Pliszka and Radek, 2017), even in highly aggressive biological environments (Skrzypczak-Pietraszek, 2015; Lipiński and Karpisz, 2020a). The use of these coatings also significantly improves the wear resistance against friction (Korzekwa et al., 2016; Szala et al., 2019), preventing contamination of the lubricants (Radzymińska-Lenarcik et al., 2018) and the associated change in material properties (Lipiński et al., 2020). The features of DLC coatings offer a chance for a significant development of implants that are more resistant to biocorrosion than the existing ones (Dudek and Włodarczyk, 2010; Wojnar et al., 2019). The methods used in the production of DLC coatings with given properties can also be useful in analogous issues of material sciences e.g. coatings for heat transfer enhancement (Orman, 2014; Chatys and Orman, 2017) and thermal comfort measure (Majewski et al., 2017), microstructure modification of oxide coatings (Korzekwa et al., 2018), SiAl alloys (Lipiński, 2015a; Lipiński, 2015b) and ferrite composites (Bochenek et al., 2018) or modification of mechanical properties (Trzewiczek et al., 2014; Lipiński and Karpisz, 2020b; Lipiński and Karpisz, 2020c). The design, manufacturing and analytical problems encountered with DLC coatings inspire the development of testing methods such as DOE (Pietraszek and Goroshko, 2014), bootstrap resampling (Pietraszek et al., 2016), quality improvement (Ulewicz, 2014), FMEA (Pacana et al., 2019) and fatigue analysis (Ulewicz et al., 2014).

Laser texturing can be used to improve tribological properties, i.e. to reduce the coefficient of friction and tribological wear of friction surfaces and to facilitate heat dissipation, increase wettability and absorptivity (Antoszewski, 2010; Dwornicka et al., 2017). Laser processing is usually carried out at power densities of 10^6 - 10^9 W/cm^2. It is based on the local effect of the laser, which causes the vaporization of the material. During this process, gas blowing (air or inert gas) is recommended to remove molten material from the cavity that may not have been vaporized. The power density and pulse duration of the laser beam have the greatest influence on the depth of the cavities (Ryk, 2002).

2. Experimental

Texturing of DLC coatings was achieved with a Trumpf TruMICRO 5325c laser with average max power 5W, pulse energy up to 12.6 μJ and beam quality M2<1.3, maximum pulse frequency 400 kHz and 343 nm wavelength .

In the first phase of the experiment, the laser radiation effect on DLC coatings was studied over a laser power range of 5 to 0.5 W and a pulse frequency range of 400 to 66 kHz at a constant processing speed of 100 mm/s.

The fabricated samples were tested on an optical profilometer to determine the depth and shape of the obtained textures. Next, the results of the tests were analyzed. The selection of appropriate parameters for the laser texturing process was guided by the shape of the texture profile, mainly its width and depth. Based on this examination, the laser erosion parameters were selected (Tokar, 2019). The texture profile made at a laser power of 3.5 W and a frequency of 66.6 kHz was selected for further study.

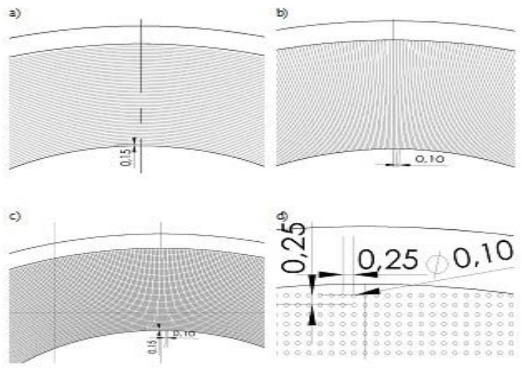

Fig. 2. Tested texture geometries: a- first, b- second, c- third, d- fourth

After selecting the suitable parameters, the texture geometry was designed in CAD program and then a driver program for the TruMicro laser was created using TruTops PFO program. Four different texture geometries have been considered. The first texture was concentric circles, each with a radius 150 μm larger than the previous one. The second one is radial lines, 100 μm apart, the third one was created by superimposing the previous two, and the fourth one are circle-shaped microcavities, 100 μm in diameter, covering the whole surface of the ring. The layout of the produced geometries is shown in Figure 2.

3. Results and discussion

The fabricated samples geometries were analyzed using a Hirox KH-8700 3D microscope. This analysis showed that the geometry of texture 1 is acceptable.

The depth of the produced grooves is about 2 μm. Analyzing the appearance of texture 2 under the microscope, one can see (as in the previous case) a complete removal of the DLC coating from the base material in the texture areas. The obtained edges of the grooves are slightly more even than in the case of texture 1. The depth of the obtained channels is about 1.5 μm. For texture 3, areas with a depth greater than the depth of the obtained grooves are formed at the overlap of the paths. As previous cases, complete removal of the diamond-like carbon layer in the laser beam passing area is observed. The depth of the microcavities was estimated to be about 1.5 μm. The edges of the cavities are uneven.

By analyzing the microgeometry of texture 4 in the form of microcircles, it can be concluded that the texturing was performed successfully. The laser beam produced microcircles with a diameter of about 100 μm and a depth similar to the previous textures, i.e. 1.5 µm. The sample on which the texture No. 4 was made was selected for further testing, i.e. microstructure analysis and tribological testing with the T-01M tester. Photographs of the fabricated textures are shown in Figure 3.

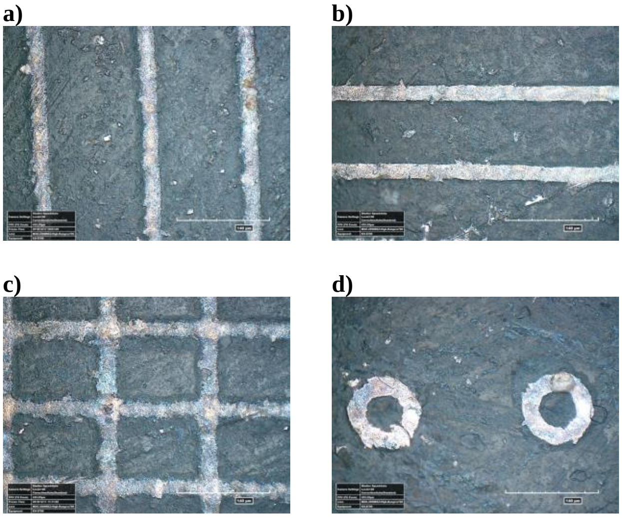

Fig. 3. Pictures of textures at 700x magnification:
a) first, b) second, c) third, d) fourth

The samples used for texturing were subjected to microhardness testing using an Innovatest Nexus 4304 hardness tester. The method used to measure hardness was the Vickers hardness method, and a 500G load (HV0.5 scale) was used to measure the microhardness of the 4H13 steel and the obtained DLC coating. The average value of the microhardness of the 4H13 steel is 631HV, while the DLC coating is 3491HV. The microhardness of the DLC coating is 5.5 times greater than the microhardness of the 4H13 steel. Coating a component made of relatively soft material with DLC coating significantly improves its hardness. Table 1 shows the chemical composition of 4H13 steel, while Table 2 shows its mechanical properties.

Table 1. Chemical composition of 4H13 steel

Elements	C	Mn	Si	P	S	Cr	Mo	V
Content %	0.45	0.50	0.60	0.04	0.03	13.5	0.5	0.2

Table 2. Mechanical properties of 4H13 steel

Tensile strength R_m [MPa]	Yield stress $R_{0.2}$ [MPa]	Elongation A_5 [%]	Hardness HRC	Modulus of elasticity E [GPa]
850-1000	650	10	52-54	215

The 4H13 steel sample coated with diamond-like carbon coating was then subjected to morphology analysis. Using the *JEOL* JSM-*7100F* scanning electron microscope with field emission, the microstructure was analyzed and a linear elemental distribution was performed along the cross section of the sample. The a-C:H:W coating was applied by PVD on a thin layer of chromium, so the elemental distributions of Cr, W, and C were analyzed (Figure 4).

By analyzing the elements distribution in the studied cross-section, a concentrated amount of chromium can be found in the middle of the scanning area. This is because in the deposition process of the diamond-like coating, a thin layer of chromium is initially applied, which significantly improves the adhesion. The linear distribution shows an increase in tungsten content at the final scanning stage. Tungsten is a component of the a-C:H:W coating applied to the 4H13 steel ring, which justifies the increase in its content. The presence of carbon atoms is also noticeable.

Tribological properties were then investigated using a ball-disc T-01M tester, where the counter specimen is a ball of 100Cr6 steel with a diameter of 6.3 mm. The ball hardness was 62 HRC. Both textured and non-textured specimens were tested for comparison purposes. Technical dry friction of the non-textured specimens was tested with the following parameters: linear speed 0.8 m/s, testing time 3600 s, for three loads 4.9 N; 9.8 N; 14.7 N. Then, the average values of the friction coefficient for each load were determined. They are summarized in Figure 5.

Currently, tests are being conducted under conditions of lubrication of friction nodes with paraffin oil.

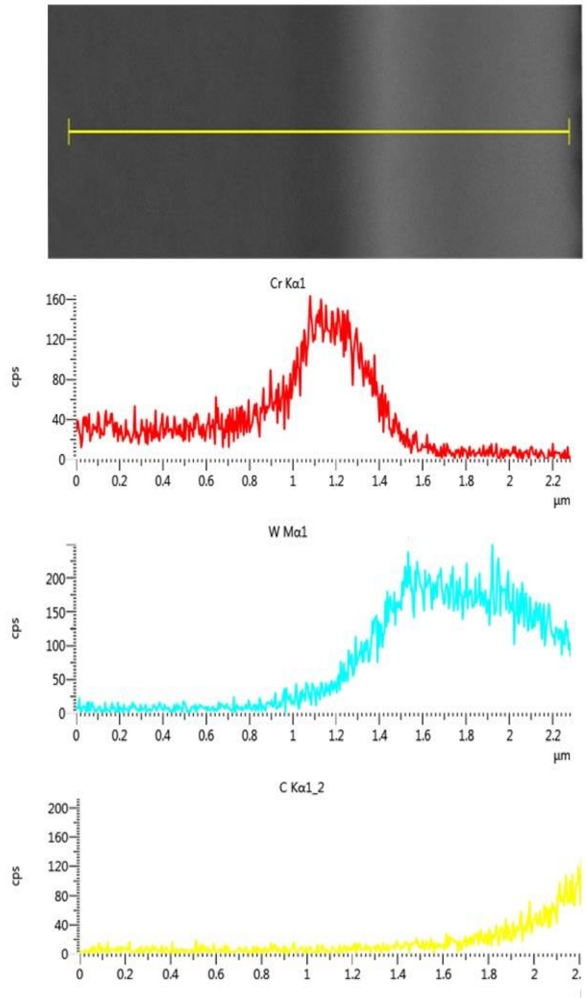

Fig. 4. Microstructure and linear element distribution in DLC coating

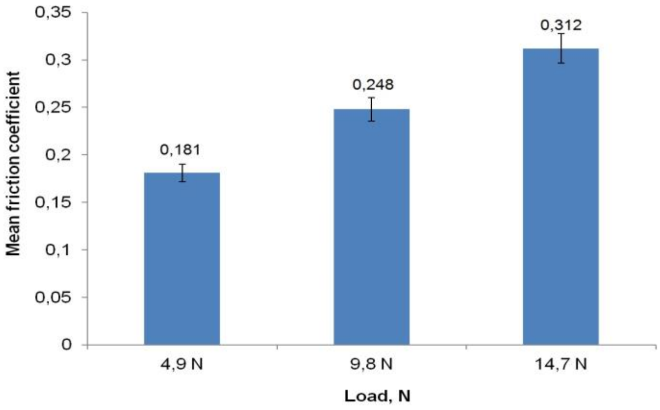

Fig. 5. Comparison of mean friction coefficient values for a non-textured DLC-coated specimen as a function of load

Analyzing the results, it is evident that the friction coefficient values increase with increasing load. The friction coefficients for the smooth DLC coating without texture remain low. The low value of friction coefficient for DLC coatings, even under technically dry friction, is their important advantage.

A comparative study of textured and non-textured specimens was also carried out with a load of 9.8 N and continuous lubrication with paraffin oil. The test time was 1000 s and the speed was 0.8 m/s. The changes in the coefficient of friction over time for the textured specimen as well as for the non-textured specimen are summarized in Figure 6.

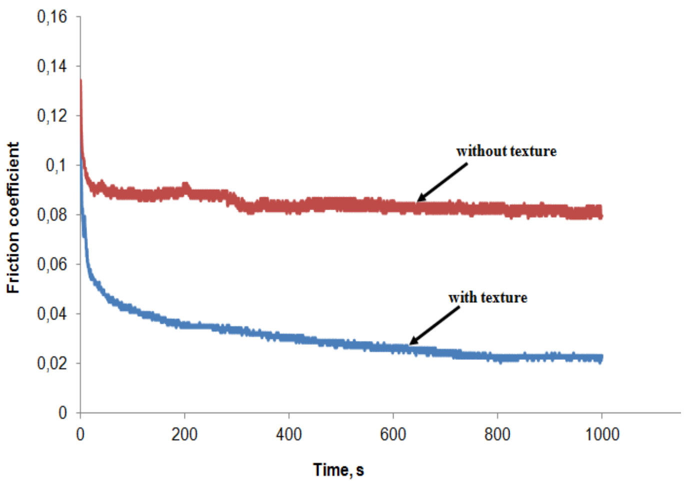

Fig. 6. Results of tribological tests of DLC coating with and without texture

4. Conclusions

1. This paper presents an tribological properties analysis of the laser-textured DLC coatings. Diamond-like carbon layers, due to their tribological properties, high hardness and biocompatibility, are widely used in technology. The test specimens were ring-shaped, made of 4H13 steel, coated with a-C:H:W layer.
2. The purpose of the study was to choose the appropriate parameters of laser radiation and to obtain textures with a specific geometry. The microhardness of the DLC coating was measured, the friction coefficients were determined, and the morphology of the sample was analyzed.
3. The thickness of the coating applied to the ring was about 1μm. It was not possible to select parameters to obtain a texture depth less than the thickness of the DLC coating. Four texture patterns were made on the samples used in the experiment As a consequence of observing the textures under magnification on a 3D microscope, the results were found to be satisfactory.
4. The analysis of the morphology and linear distribution of elements in the transverse section of the specimen confirmed the method of deposition of this layer. The presence of chromium was detected in half of the scanned area. This element is applied to the coated material to increase the adhesion of the coating. Significant amounts of carbon and tungsten components of the a-C:H:W coating were found in the final scanning area.
5. Microhardness tests were performed and a 5.5 times higher hardness of the diamond-like carbon layer with respect to the substrate material was reported. The application of diamond-like carbon layer as a coating layer on components made of materials with lower hardness significantly improves their functional properties.

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激光纹理化对 DLC 涂层的摩擦学性能的影响

關鍵詞
DLC
激光纹理化
涂料层 摩擦学性质
石墨

摘要

这项工作提出了使用 DLC 涂层的激光织构化以改善摩擦学性能的方法。 通过 PVD 方法将涂层施加到由 4H13 钢制成的环上。 用 TruMICRO 5325c 皮秒激光器对表面进行纹理处理，辐射波长 · = 343 nm。 进行了表面微观结构分析，表面微观几何形状和显微硬度测量以及摩擦学测试。本文中提出的问题可用于扩展 DLC 涂层应用领域的知识，尤其是在滑动摩擦副中。