Parameters of protective carbon films applied on high-speed steels M42 via magnetronic sputtering

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High-speed steel M42, which is used as material for saw teeth in the most popular bimetallic saws, is considered as one of the most universal high-speed steel grades. This steel is characterized by high hardness (HRC 64-67), excellent heat resistance up to 700 °C, good wear resistance and is suitable for cutting of stainless steels with hardness up to 50 HRC. Nanostructured wear-resistant high-hard coatings with high physical and mechanical properties, resistance to impact and vibration loads promotes increase of wear resistance and service life of band saws. The films of hard diamond-like carbon (DLC), containing amorphous carbon with disordered graphite ordering, which is striving to a tetrahedral diamond-like coordination, are interesting for material scientists due to their excellent mechanical and tribological properties. Dense and hard carbon films with structure of nanoparticles having size 12-17 nm, were applied on high-speed steel M42 (according to AISI) via the method of high-frequency magnetronic sputtering. Microhardness of DLC film of high-speed steel M42 (M42 HSS) by Vickers with the coating thickness 800 nm was 838±19 in comparison with 783±18 for a clean sample of the same steel. Good microstructure, wear protection properties and high temperature oxidation protection of obtained carbon nanostructured films on the surface of M42 HSS are observed.
Key words: magnetronic sputtering, wear, high-temperature protection, coating, nanostructured films, high-speed steel.
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Introduction

Metal-cutting disk and band sawing machines for cutting of rolled products and tubes from ferrous and non-ferrous metals are used intensively at present time. M42 high-speed steel is the most universal grade of such steels which are used as material for teeth of the most popular bimetallic saws [1]. This steel is characterized by high hardness (HRC 64-67), excellent heat resistance up to 700 °C, good wear resistance and is suitable for cutting of stainless steels with hardness up to 50 HRC. Such saws are used for cutting of majority of steels, including usual stainless steels. Thus, a saw tooth made from M42 steel is suitable for cutting of usual ferrite stainless steels, meeting the requirements of AISI 430 SS.

Nanostructured wear-resistance coatings with high hardness and high physical and mechanical properties as well as with high resistance to impact and vibration loads promotes increase of wear resistance and service life of band saws [2, 3]. The films of hard diamond-like carbon (DLC), containing amorphous carbon with disordered graphite ordering, which is striving to a tetrahedral diamond-like coordination, are interesting for material scientists due to their excellent mechanical and tribological properties. Carbon films of DLC type present a metastable mixture of solid phases – hard diamond-like phase with sp^3-links and more soft graphite-like phase with sp^2-links [4, 5]. Relationship between compositions of two carbon phases in obtained DLC film depends on the method of deposition and different parameters. Multiple experimental results showed that amorphous DLC films are usually characterized by low friction coefficient, high hardness, good chemical inactivity, optical transparency in the infrared range, excellent service life, and they are used as wear-resistant and corrosion-resistant coatings [6]. DLC film displayed good friction lowering and good wear-resistance properties during slipping along steel analogue materials in lubrication-free conditions; it was explained by DLC film graphitization and forming of transferred DLC layer on the surface of steel analogue material [7]. Carbon films, which were obtained via magnetronic sputtering in the direct current conditions [8-10] and via high-frequency non-balanced magnetronic spaying [11] were examined previously. Thereby, examination of surface structure and surface quality for amorphous carbon films, with were applied by magnetronic sputtering in the high-frequency direct current conditions with different thickness on M42 high-speed steel substrate, was the aim of this work. The main attention in this investigation was paid to microstructural, thermal emission, micro-hard and tribological properties of obtained carbon-base films.

Materials and equipment

Thin hydrogen-free carbon films with thickness from 100 to 800 nm were applied on substrate from high-speed tool M42 steel (AISI) using high frequency (13.56 MHz) via magnetronic sputtering from graphite target (Quorum Q150 ES). 2.25 inch toroidal magnetronic system with high frequency capacity 85 Wt was also used. High pure pressed graphite foil (nominal purity > 99.995 %) was used as the aimed carbon source. Argon with nominal purity > 99.998 % was used as a plasma-forming spraying gas.

Thin 2 mm samples of polished substrates from M42 high-speed steel was used as substrate material. These steel samples were purified in an ultrasonic sink by acetone and ethanol consequently, and then they were dried in the vacuum furnace DZF-6090. The procedure of argon-plasma pickling was used for purification of substrates with removal of residual surface contaminations. To provide sputtering, pressure in the chamber was decreased to basic value $2x10^{-3}$ mbar. As a result of argon introduction in the vacuum chamber, pressure was about 10^{-2} mbar. Respectively, the substrate temperature was controlled during spattering process using electrically isolated thermocouple of K-type, which was mounted on the rear side of substrate holder.

At small thickness of sputtered carbon films, substrate temperature ~36±2 °C was observed, while maximal temperature (~ 43 °C) was registered for sputtering with maximal energy and time consumption; then thin carbon films with thickness 800 nm were obtained. Sputtering was conducted at the room temperature, processing time for such film constituted up to 3.7 hours. Distance between substrate from M42 steel and spraying graphite target was fixed at the level 60±1 mm.

Topography of thin sputtered DLC films with different thickness was researched using scanning electron microscope (SEM) JEOL JSM-7500F with accelerating voltage 15 kV and working distance 7.6 mm in deep vacuum.

Spectra of optical diffusion reflection of sputtered carbon films on the surface of M42 high-speed steel were measured by a spectrophotometer Hitachi U-3900 with dual channel integrating sphere. The values of electronic permitted area E_g from optical measurements of diffusion light reflection were determined via Kubelka-Munk equation.

$$F(R) = \frac{(1 - R)^2}{2R} \quad (1)$$

Influence of volumetric hardness of carbon films from M42 steel with coating were checked by Falcon 500 microhardness meter via applying the load 0.05 kgf by a pyramidal micro-indentor to a coated surface and consequent holding during 15 s. Then load was removed slowly and print square was calculated via measurements of diameters by a microscope with minimal step 0.01 mm. The measured hardness values for three different tests ($HV_{0.05}$) were used afterwards.

Tribological measurements of formed DLC coatings with thickness 600 nm were carried out on the samples from M42 high-speed steel using an oscillating tribometer MMDY of “pin on disk” type, which operates with metallic ball pin from stainless steel AISI 430, diameter 5 mm and normal load 2 N. Steel ball (AISI 430) with diameter 5 mm and average hardness 228 ± 10 HV is used. A pin makes oscillations along the sample surface with amplitude difference 0.5 mm and frequency 5 Hz. Experiments were conducted in closed atmospheric conditions at permanent room temperature 23 °C and air humidity 65±5 %. Friction between pin and sample initiates sample oscillations, which are registered for evaluation by a friction coefficient. Central section of each wear path was measured by digital profilometer TR100. Micro-scales Mettler Toledo XP6 Automated-S were used for determination of density of manufactured DLC film.

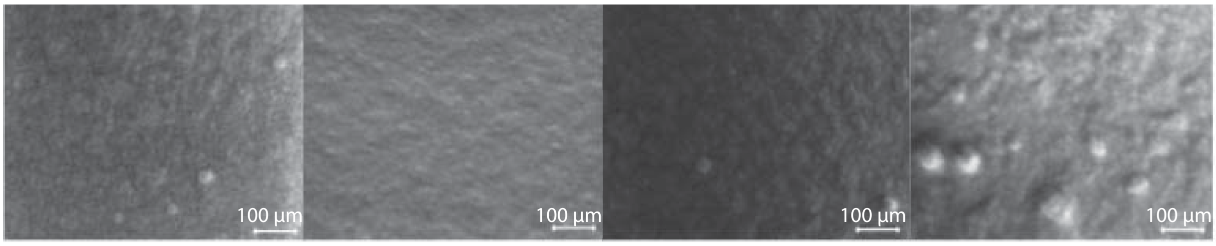
Digital micrometer UT620A was used for measurement of surface electric conductivity of thermally annealed samples of high-speed M42 steel in pure form and with DLC coating films. The heat emission coefficient of pure, oxidized and annealed substrate from high-speed M42 steel with DLC coating was determined by digital thermometer UT303D with evaluation of heat emission coefficient via averaging of the results of three independent measurements, its measuring accuracy made 0.01 rel. units. Electric hot plate IKA C-Mag HS7 with metalloceramic heating surface with digital control was used for heat treatment of samples with pure and DLC-coated substrates from high-speed M42 steel with heating rate 50 K/min and stabilization during 2 min at 300 °C. Thermal camera Smart Sensor ST9450 with operating range of wave length 8-14 μm was used for observation on temperature fields on the surface of researched samples from high-speed M42 steel.

Obtained results

Obtained thin carbon films were dense and were applied well on metallic and glass substrates without micro-holes and have practically sufficient adhesion degree, which was determined qualitatively on researched metallic surfaces on the base of scratch test via the method of quartzite pencil and the method of adhesive tape tearing. High-frequency magnetronic sputtering with density of capacity 2.2 Wt/cm^2 led to average speed of carbon film growth 2.4±0,2 nm/min. To achieve more high growth speed, density of capacity on graphite target was increased step-by-step up to 3.3 Wt/cm^2, what was finalized in average experimental speed of thin carbon film growth 3.6±0,2 nm/min.

Obtained carbon films present amorphous carbon structures according to SEM, these structures are slightly alloyed by nitrogen with its calculated content about 4±1 % (atom.) in correspondence with measurements of quantitative analysis of electronic diffraction structure (EDSA). Small nitrogen impurity in a sputtered carbon film was probably connected with argon impurity as plasma forming gas and with incomplete pumping in the process of magnetronic sputtering.

Based on the SEM data, it is clear that fabricated carbon coatings contain clusters of nano-particles with size 12-17 nm, and size of these clusters is slightly varied in carbon coatings with different thickness. Isolated small islands with size about 60-80 nm were seen in separate areas of the obtained carbon film according to the previous researches (**Fig. 1**) [12]. It should be underlined that serious increase of the part of island agglomerated particles takes place for film thickness 800 nm.



a *b* *c* *d*

Fig. 1. Microstructure of sputtered carbon coatings:
a – 200 nm; *b* – 400 nm; *c* – 600 nm; *d* –800 nm

Table 1. **Parameters of applied carbon films**

Film thickness, nm	S_a, nm	R_a, nm	$D(м)_{av}$, nm	$D(z)_{av}$, nm	S_{sk}
200 нм	1.07±0.10	0.93±0.08	9.10±1.99	13.04±2.38	1.183
400 нм	0.99±0.10	0.69±0.06	10.76±2.54	12.52±1.98	1.268
600 нм	0.88±0.09	0.66±0.09	11.02±2.38	13.35±2.18	1.423
800 нм	1.76±0.12	1.45±0.16	14.93±2.47	14.47±3.79	1.439

Average roughness values of examined films obtained via the method of S_a torques analysis, average roughness values on lines and columns of R_a image, average diameter of particles via the method of $D(m)_{av}$ torques analysis, average diameter of particles $D(z)_{av}$ via the method of analysis of roughness parameter R_z, coefficient S_{sk} of shape asymmetry of particles were calculated using computer program Gwyddion 2.54 and presented in the **Table 1**.

According to the data of the Table 1 it can be concluded that the average size of nano-particles in carbon film increases systematically with rise of thickness of this film, which is applied by magnetronic sputtering; the coefficient of shape asymmetry of particles increases as well. Calculated average roughness (S_a и R_a) at first decreases with rise of thickness of carbon film, but increases for the film with thickness 800 nm. It is probably caused by appearance of the serious part of agglomerated island clusters of nano-particles (see Fig. 1*d*).

The values of fractal dimension *D* (dimension of Hausdorf-Bezikovich), approximated by the cube counting method and triangulation method, were calculated for obtained nano-structured carbon coatings using computer program Gwyddion 2.54 and presented on the **Fig. 2**. The value *D* will be equal to 2 for absolutely flat square or round surface without any ledges, while for the surface with maximal possible roughness presented by endless multitude of vertical peaks this value will approximate to 3.

According to the data on the Fig. 2*a*, calculated fractal dimension in fabricated carbon coatings is high and it reduces systematically with increase of carbon layer thickness. Observed decrease of fractal dimension in the obtained nano-structured carbon films takes place owing to recrystallization of round carbon nano-particles in more flat ones in the durable process of magnetronic spraying. It is known that surface quality has significant effect on such operating properties of products as wear resistance as well as tribological and optical reflecting properties [13]. Decrease of fractal dimension in obtained thick carbon films can be interpreted as appearance of carbon nano-particles with more flat planes, what should promote wear resistance rise and lowering of friction coefficient of these films.

Data of the Fig. 2*b* display that used nano-structured carbon coatings also increase microhardness of working surfaces of the HSS M42 steel sample as a model of saw teeth. Thus, the obtained results of electron microscopy and microhardness measurement show that the developed single-layered carbon nano-structural coatings with low roughness increase apparently hardness of working surfaces of a band saw made of high-speed steel M42.

Calculated values of width of electronic prohibited area Eg for obtained thin carbon films with thickness from 100 to 800 nm is situated within the range 4.23-4.30 eV, and they are typical for dielectrics. Such obtained E_g values are below 5.5 eV for pure diamond [5], but higher than the range 3.37-3.68 eV for DLC film from [14] and higher than E_g value equal to 2.55 eV for films of polymer-like carbon (PLC) [15] or than E_g values within the range 1.6-2.1 eV for films of amorphous carbon, which were deposited via the method of impulse non-balanced magnetronic spraying [15]. It can be suggested that a mixture of different carbon phases with prevailing of wide-zone diamond-like phase with sp^3 hybridization is presented in the obtained carbon nano-structured films.

Micro-weight measured and calculated average mass density of obtained DLC coatings with layer thickness 800 nm makes 2.8±0.2 g/cm^3, what exceeds the value 2.2 g/cm^3 for graphite-like amorphous carbon films [3] and which is smaller than the value 3.2 g/cm^3 for tetrahedral amorphous carbon [4]. Thereby it can be concluded that strongly distorted cellular structure of carbon films with prevailed sp^3 links and essential part of tetrahedral amorphous carbon phase was obtained in the process of sputtering. This conclusion agrees with another conclusion about

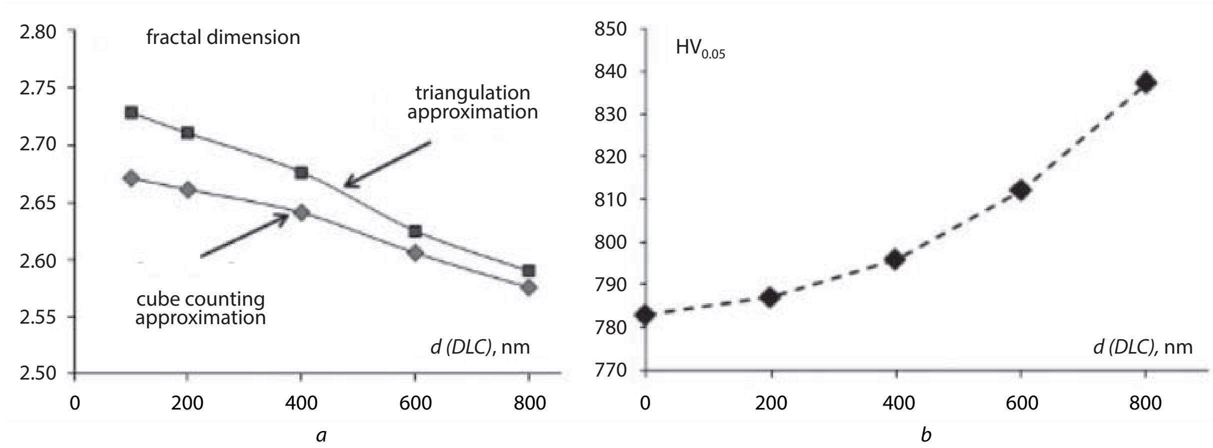


Fig. 2. Fractal dimension in nano-structured carbon coatings (*a*) and Vickers microhardness of the initial material samples of high-strength steel M42 with applied nano-structured carbon coatings (*b*)

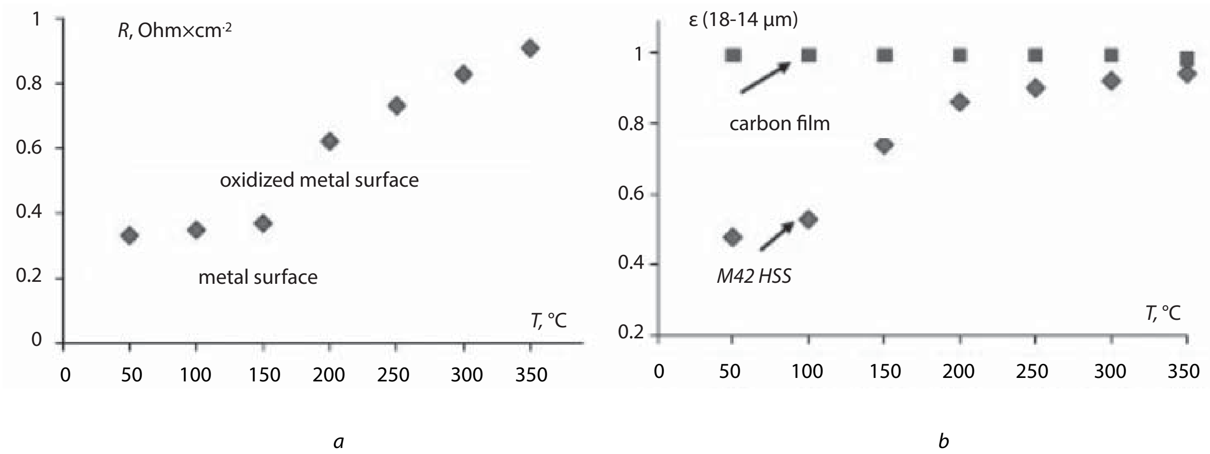


Fig. 3. Temperature relationship of surface electric resistance of clean M42 steel samples (*a*) and heat transfer coefficient (*b*) for clean M42 steel and M42 steel with applied nano-structured carbon coatings

mixture of carbon phases on the base of analysis of the prohibited area width in applied carbon coatings.

Spectral emitting capacity describes material surface ability to provide emission of photons within the thermal range on the definite wave length relating to emission of the perfectly black body. It is determined as relation between radiating energy, which is emitted by a real object, and energy, which is emitted by the black body on the same wave length and at the same temperature. It is known that observing thermal emitting capacity (heat transfer coefficient) in measuring metals increases with rise of their surface roughness [16. 17]. Numerous experiments confirm that both surface oxidation and surface roughness have influence on spectral emitting capacity, and surface oxidation provides more dominating influence [16, 17].

Then the results of investigation of influence of oxidizing annealing in the air on parameters of nano-structured carbon coating for a sample of high speed M42 steel are examined. Accelerated thermal annealing with rate 50 °C/min and holding during 2 min in the interested temperature points was conducted for samples of simulated saw teeth from high speed M42 steel with carbon coating and without it. The temperature range of annealing was 50-350 °C with step 50 °C.The heat transfer coefficient was determined three times during 20-30 s at each temperature after heating during 2 min (**Fig. 3**) in normal direction to a saw tooth, which was fixed horizontally on ceramic surface of electric stove heating surface.

Systematic increase of observed parameters of the surface electric resistance for the samples of high-speed M42 steel and the values of heat transfer coefficient in the medium infrared range is connected with forming of metal oxides films on the surface of this steel. While conducting thermal oxidizing annealing of high-speed M42 steel up to the temperature 350 °C, the following metal oxides are most possible as low-temperature oxidation products: Fe_3O_4 and Fe_2O_3, MoO_2, VO_2, Cr_2O_3, Co_3O_4. In this case M42 steel has such composition: Mn 0.5 %, V 1 %, Co 1 %, Cr 4 %, Mo 10 %, Fe >82 %. The above-listed oxides are classifies as semiconductors and respectively have more high heat transfer coefficient in the medium infrared range. Increase of roughness of surface oxide films, which are forming dur-

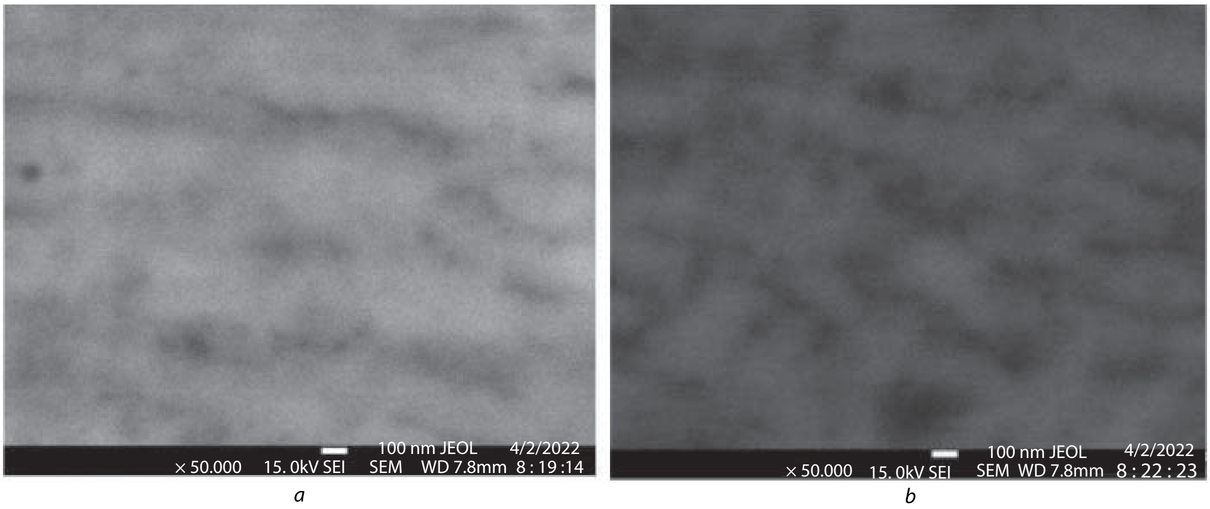


Fig. 4. Microphotograph of M42 clean steel sample surface before (*a*) and after (*b*) thermal annealing at the temperature 350 °C

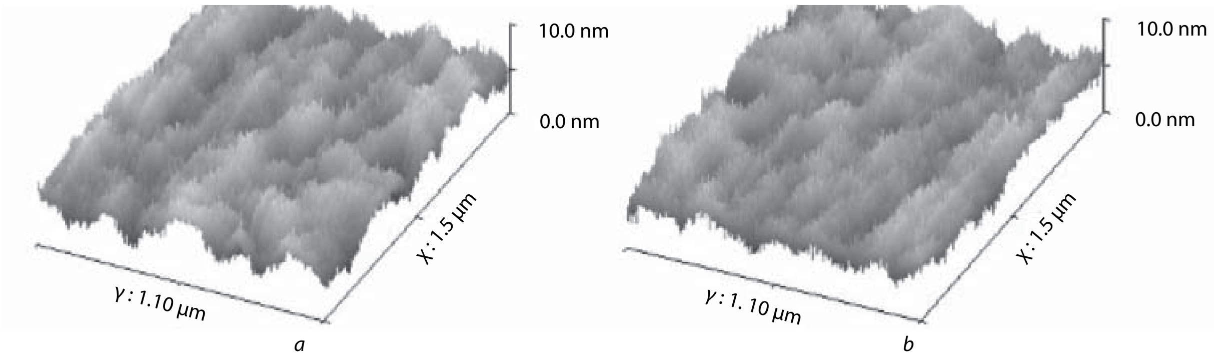


Fig. 5. Calculated topology of the surface of M42 steel sample surface before (*a*) and after (*b*) thermal annealing at the temperature 350 °C

ing oxidizing thermal effect on the surface of high-speed M42 steel, should also lead to rise of infrared thermal emission parameters of annealed sample of high-speed steel with temperature elevation.

The results of investigation of temperature influence within the range 100-350 °C on microstructural and spectral physical properties of M42 steel displayed that its surface topology , microstructure and surface roughness vary substantially in the process of forming of surface wide area semiconducting oxide films (**Fig. 4** and **5**).

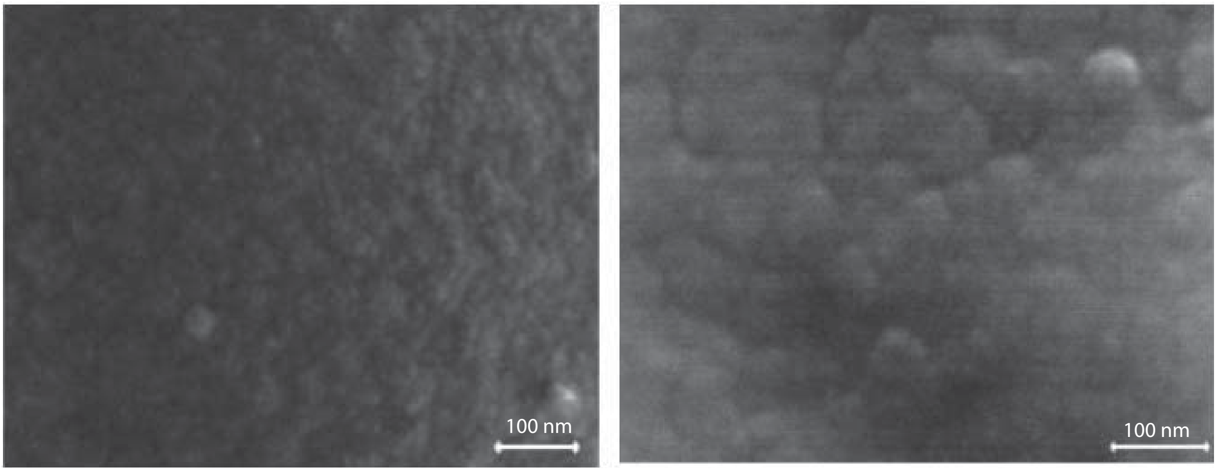
According to the Fig. 4 data processing, average roughness Sa of high-speed M42 steel increases during thermal annealing from 0.72±0.04 to 0.95±0.05 nm, what testifies on essential influence of annealing on microstructure of steel surface owing to forming of surface oxide films.

It should be also noted, that heat emission parameters for developed nano-structured DLC-like wear-resistant coatings on the surface of cutting edge of band saw teeth from high-speed M42 steel in the infrared range did not vary practically within the temperature range 50-350 °C. It testifies on high temperature oxidizing protective properties of obtained nano-structured film and allows to provide efficient heat removal from the surface of high-speed M42 steel.

Microphotographs of the surface of high-speed M42 steel with carbon film with thickness 600 nm, which were made before and after stadial (step-by-step) thermal annealing at the temperature 350 °C, are presented on the **Fig. 6**.

According to the processing of the data from Fig. 6, average roughness Sa of carbon film with thickness 600 nm increases from 0.88±0.09 to 1.08±0.14 nm during thermal annealing of M42 steel with this film; it testifies about influence of annealing on surface microstructure of protective carbon film. Supposedly, thermal annealing of M42 steel with carbon film with thickness 600 nm is accompanied by collective recrystallization of nano-structured carbon film with forming of large amount of agglomerated clusters of nano-particles.

Friction coefficient and wear profile for HSS M42 steel with and without protective carbon coating were determined on the base of measurements of tribological properties. Friction coefficients for HSS M42 steel in comparison with stainless steel SS430 are located within the range 0.49±0.01



a *b*

Fig. 6. Microphotograph of M42 clean steel sample surface with carbon film with thickness 600 nm before (a) and after (b) thermal annealing at the temperature 350 °C

for a non-oxidized sample and 0.57±0.02 for a steel M42 sample, which was oxidized at 300 °C. It was found out experimentally that friction coefficient of the applied DLC coatings, slipping on a ball of stainless steel SS430, is stable within the range 0.11-0.13 and then lowers to 0.08 for the large number of cycles (38,000-40,000). Thus, the average value of friction coefficient made FC=0.11±0.02 for the samples of M42 steel with applied DLC coating in comparison with SS430 steel. This result correlates well with previous researches of tribological parameters of DLC films [18, 19]. The authors suggested that lowering of friction coefficient for DLC coatings, slipping on stainless steel SS430, is observed owing to the process of thermomechanical induced graphitization, which is caused by running large distance in slipping conditions. Graphitization of the surface of DLC films is probably caused by stresses arisen by friction contact (not by heat as a result of friction), because temperature rise in tribological testing is rather slight.

Observed stability of the value of friction coefficient for the developed diamond-like coating and behaviour of wear traces after oxidizing thermal annealing at 350 °C for a processed sample of high-speed M42 steel attracts attention and probably testifies on sufficient practical stability of corrosion-resistant carbon nano-structured coating at increased temperature.

Obtained DLC coating at M42 steel plates have low friction coefficient, what provides definite advantage during dry metal processing both alloy steels and non-ferrous metals. It is known that DLC films on the surface of chisel lathe plates are characterized by good adhesive resistance within the temperature range up to 400-450 °C, what provides excellent removal of flow spiral chips from non-ferrous metals without its twisting on billet body or chisel plate. In the case of mechanical processing of oxidized surface of heat treated alloy steels, low friction coefficient of DLC film allows to remove oxide layer with lower energy consumption and less heat extraction.

According to the data from the work [20], wear of metal processing tool occurs owing to the complex effect, including mechanical, thermal and chemical effect on material surface. The wear mechanisms of lathe plates include adhesive, diffusive and oxidizing wear [20]. DLC films, owing to low friction coefficient COF <0.2, can decrease efficiently both abrasive and diffusive wear due to lowering of heat extraction. Extremely high chemical resistance and high oxidizing heat resistance of DLC films at the temperature up to 500 °C [21] decreases strongly possibility of adhesive and oxidizing wear of teeth of band and circular saws and lathe chisels. It is also necessary to take into account that DLC films have very high heat emission coefficient (heat exchange coefficient), about 0.99-1.00 in thermal infrared range (8-14 μm) in comparison with heat emission coefficient for metals (0.1-0.4). It allows to provide efficient heat emission from the surface which is coated by DLC film, or from a saw tooth of chisel plate, via heat emission during mechanical processing of metals.

According to the respectable opinion of the specialists of Sumitomo company, metal processing tools, which are coated by protective film presented by carbon DLC coating, has smaller cutting force in comparison with usual hard alloy tool (both in wet and dry conditions). For example, the above-mentioned specialists consider that decrease of the required pulling force during drilling processing of alloyed aluminium alloys by 67 % using tool with DLC coating provides higher drilling efficiency in comparison with tools without coating and with deeding speed larger by 3-5 times. Decrease of cutting force by 45-46 % when using tools with DLC coating also reduces substantially surface roughness of alloyed aluminium alloys [22], avoiding necessity in the most part of additional operations for burrs removal.

The specialists also emphasized one other essential advantage of DLC coating on lathe and milling tools: it is their increased service life. Tools with DLC coating display increase of service life for metal processing tools in average by 119 % in the case of processing of alloyed aluminium alloys (in comparison with other competitive tools). It is also noted that increase of service life of lathe tool by 50 % can lead to lowering of metal processing cost by 1/3 [23]. It was shown earlier that testing of lathe processing of Al-Si alloys using lathe plates from cemented carbides of K 20 grade displayed reducing of the required feeding force by 11.6 % (for the alloy with 12 % (mass.) of silicon) and by about 16.3 % (for the alloy with 16 % (mass.) of silicon), when using tools with DLC coating [24]. It was also noted above that use of protective DLC coating on hard alloy WC-Co chisels in lathe processing of fiberglass [25] allows to increase service life of a chisel in its side wear almost by 4 times, what provides reducing of lathe processing cost more than by half.

Conclusions

This work showed that solid amorphous carbon films with different thickness (from 100 to 800 nm) can be sputtered via the method of high frequency magnetronic spraying in the conditions of low density of fed power, providing essential content of sp^3-fraction of diamond-like carbon. The properties of microstructure of obtained carbon films on the surface of high-speed steel M42 were examined and their expressed nano-structured properties with average size of nano-particles from 9 to 15 nm and low roughness were shown. Use of obtained carbon films for wear protection and high-temperature oxidation of HSS M42 steel as material for circular and band saws was predicted on the base of thermal stability, heat emission and tribological tests.

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