Archives of Materials Science and Engineering

Volume 54
Issue 2
April 2012
Pages 60-67

International Scientific Journal
published monthly by the
World Academy of Materials
and Manufacturing Engineering

Optimization of the deposition parameters of DLC coatings with the MCVA method

M. Pancielejko*, A. Czyżniewski, V. Zavaleyev, A. Pander, K. Wojtalik
Institute of Mechatronics Nanotechnology and Vacuum Technique,
Koszalin University of Technology, ul. Śniadeckich 2, 75-453 Koszalin, Poland
* Corresponding e-mail address: mieczyslaw.pancielejko@tu.koszalin.pl

Received 09.02.2012; published in revised form 01.04.2012

ABSTRACT

Purpose: The purpose of the present study was to determine the optimal values of selected deposition parameters of diamond-like-carbon coatings (DLC) with the modified cathodic vacuum arc (MCVA) method which ensure obtaining of their most advantageous properties from the perspective of their application for the coating on high-speed steel tool substrates for woodworking.

Design/methodology/approach: An analysis was conducted of the investigations into the influence of the selected deposition parameters of DLC coatings on the accepted optimization criteria with the use of the Taguchi module. Adhesion, hardness and friction wear resistance were accepted as the optimization criteria of DLC coatings for high-speed steel substrates.

Findings: It was established on the basis of the statistical analysis of the research results that in order to ensure a high adhesion of DLC coatings to high-speed steel substrates, a thick Cr sublayer (0.3 μm) and a DLC coating (1.8 μm) is to be used, which is deposited at a high argon pressure (0.25 Pa); no substrate bias (the floating potential) is to be used. In order to obtain high hardness and friction wear resistance, higher values of substrate bias voltages (-80 V) and a low pressure of argon (0.01 Pa) are to be used.

Research limitations/implications: To evaluate with more detail the possibility of applying these coatings on tools. I will be kept industrial tests of wearing out tools covered with these DLC coatings.

Practical implications: The properties of DLC coatings that are deposited with optimized parameters may indicate the possibility of their application for woodworking or tools for wood-like materials in order to increase their durability.

Originality/value: From results of the optimization of selected deposition parameters of DLC on the Taguchi method is possible to appoint coating properties. Depending of the deposition parameters applied, it is possible to obtain DLC coatings in a wide range of hardness (20-60 GPa). The DLC coatings might be apply on high-speed steel knives for woodworking industry.

Keywords: Tool materials; Diamond-Like-Carbon coatings; Modified cathodic vacuum arc; Taguchi optimization method

Reference to this paper should be given in the following way:

M. Pancielejko, A. Czyżniewski, V. Zavaleyev, A. Pander, K. Wojtalik, Optimization of the deposition parameters of DLC coatings with the MCVA method, Archives of Materials Science and Engineering 54/2 (2012) 60-67.

PROPERTIES

1. Introduction

In spite of the common application of hard of and super hard machining materials, steel tools are still used due to a high ductility and fracture toughness [1]. A modification of tools produced from tool steels and from sintered carbides consists chiefly in the deposition with the physical vapour deposition methods (PVD) on their working surfaces of thin single and multilayer coatings, nitrides and carbides of transition metals (Ti, Cr, W) as well as single-component and multi-component ones with the additions of light elements (Al, Si) [2-8]. Carbon based coatings are increasingly more frequently used [4,5,8-12]. There are many classifications of diamond-like-carbon coatings (DLC), which include according to [10,13-15]: a-C - amorphous carbon, ta-C - tetrahedral amorphous carbon, ta-C:H - hydrogenated tetrahedral amorphous carbon, a-C:H:Me - nanocomposite modified with metals (Me - Ti, W, Mo, Nb), a-C:H:MeC - nanocomposite modified with metal carbides (Me - Ti, W, Mo, Nb). DLC coatings exhibit such properties which indicate a possibility of their use for the coating of woodworking tools and tools for wood-like materials. They possess a high hardness of over 50 GPa [13,16,17] and a high friction wear resistance [12,17,18]. A graphitization of a DLC coating under the influence of the temperature and stresses may result in a reduction of friction resistances [10,17,19]. They exhibit chemical inertness which gives a high corrosion resistance in water environment [5]. They are thermally stable in high temperatures, frequently over 600°C [10,14]. In the deposition of DLC coatings with the use of PVD methods, a low temperature of the process below 200°C is possible [3,13,16-20].

The investigations presented in the study concerned those DLC coatings which constitute a mixture of the ta-C phase and the graphite-like phase - graphitic C [13-15]. The properties of these coatings depend chiefly from the energy of ions during their deposition, which can be affected in a wide range through the change of the substrate bias and the argon pressure [13-15,20,21]. The thickness of the DLC coating has an influence on internal stresses in the coating, whereas the use of a metallic intermediate layer may result in a reduction of stresses in the system of the substrate - the DLC coating and an improvement of adhesion [4,9,13,22].

The purpose of the present study was to establish optimal values of selected deposition parameters of DLC coatings with the use of the modified cathodic vacuum arc method, which guarantee obtaining their most favorable properties from the perspective of their application for the coating of HS6-5-2 high-speed steel tools for woodworking. An analysis was conducted of the results of the investigations of the influence of the selected deposition parameters of DLC coatings on the accepted optimization criteria with the use of the Taguchi module in STATISTICA version 9.0 program, StatSoft, Inc. 2009. Adhesion, hardness and friction wear resistance were accepted as the optimization criteria of DLC coatings for HS6-5-2 high-speed steel substrates.

2. Experimental

2.1. Deposition procedure

The coatings were deposited in a C55CT technological installation manufactured by INOVAP Dresden [23], which was located at the Institute of Mechatronics, Nanotechnology and Vacuum Technique in the Koszalin University of Technology. The vacuum chamber possesses two arc sources with graphite targets (ø 72 mm and a thickness of 10 mm, with the purity of 99.99%) and one source witch chromium target (with the purity of 99.9%). The modified cathodic vacuum arc method (CVA) was used, which consists in applying on the direct current ground coat with the current value of 50A a pulse discharge with a sinusoidal shape and the maximum current intensity value of 1400-1600 A. The frequency of the pulse repetition was 100 Hz, and its duration was ca. 0.3 ms. The technique used results in a substantial increase of the plasma ionization degree and may have an influence on an improved adhesion of carbon coatings with a relatively low deposition temperature [16,23]. Table 1 includes the main technological parameters of the C55CT installation.

The coatings were deposited on the following set of substrates: HS6-5-2 high-speed steel (dimensions 30×30×3 mm), after heat treatment (hardness: 62-63 HRC) and grinding (Ra < 0.1 μm) and polished silicon plates (type: n, dimensions 30×5×0.5 mm). Prior to deposition, the process consisted of ultrasonic-aided cleaning with detergents and extraction naphtha, acetone and ethanol were applied to the high-speed steel substrates. Once the substrates were placed in the chamber and the pressure of residual gases reached ca. 10^{-3} Pa, cleaning was applied with chromium ions with the substrate bias voltage of -580 V in an argon atmosphere with the pressure of ca. 0.2 Pa. Next step thin chromium interlayer was deposited and for the end the DLC coating was deposited. A planetary system was used for the rotating of the elements being coated.

Table 1.
Technological parameters of C55CT installation

Parameter	Range
Direct current discharge - Current intensity - Voltage	 50-100 A 15-20 V
Current pulses - current intensity (peak value) - voltage - frequency of pulse repetition - duration of pulses	 1400-1600 A 80-85 V 100 Hz ~0.3 ms
Pressure - initial - working	 10^{-3} Pa do 0.5 Pa
Maximum temperature	< 200°C
Substrate bias voltage	-580 V - floating potential
Process gases	argon, nitrogen, acetylene
Distance between target - substrate	~ 190 mm

2.2. Experimental procedure

The thickness of the DLC coatings was established on the silicon plates by means of the profilographometric method on a Hommel Waveline T8000 device and on the basis of an observation of the fractures of the coatings with a SEM - JEOL 5500 LV scanning electron microscope (SEM). The chemical composition of the coatings was examined by means of an EDS - INCA x-ray microanalysis.

The adhesion of the coatings to the substrates was characterized in a scratch test with a Revetest® Scratch-Tester devices manufactured by CSM Instruments, Switzerland. Critical load L_c is the measure of adhesion which causes an adhesive damage of the coating. The following settings of the device were used when making the measurements: load changes in the range of 0-100 N; normal loading rate 100 N/min; relative travelling speed of the table with the sample 10 mm/min; length of the scratch made 10 mm or 5 mm; distance between successive scratches ca. 1 mm. It is evident from the conducted microscopic optical analyses of the nature of damages to DLC coatings that they are subject to adhesion damage usually on three stages. With L_{c1} critical load there occur single small splinters or damages to the coating of a cohesive nature. With L_{c2} critical load, there occur distinct and regular damages to the coating; with L_{c3} critical load, there usually occurs a total rupture of the coating. The adhesion of the coatings was also described by means of Rockwell tests which consisted in driving in an intender according to scale C and a determination of the nature of damages to the coating that was expressed in HF1-HF6 degrees.

The hardness (H) and the modulus of the elasticity of the coatings were determined by means of the loading-unloading method with the use of a Berkovich indenter on a FISCHERSCOPE HM2000 XYp microhardness tester with an indentation depth below 10% of the thickness of the coatings.

Tribological tests of the coatings were conducted on a T01M tester according to the ball-on-disc method. Al_2O_3 ceramic balls (ø10mm) were used as counter-samples. For the purpose of a frictional contact of the DLC coating with an Al_2O_3 counter-sample, the following test parameters were used: normal load $L = 20$ N; sliding speed $v = 0.2$ m/s; friction radius $R = 10$ mm. The tests were carried out in ambient air of relative humidity 40-50% and temperature 22-23°C. The volume of the worn coating was determined. Further, the rate of the frictional wear of coatings (k_{vA}) was determined from the Archard's equation according to the procedure which was described among others in the papers [18,22,24,25].

2.3. Optimization method of deposition parameters

An analysis was conducted of the results of investigations into the influence of the selected deposition parameters of DLC coatings on the accepted optimization criteria with the use of the Taguchi module in Statistica program. Signal to noise ratios (ETA) were used that were proposed by Taguchi depending from the required properties of a DLC coating: "the greater the better" - used when it was expedient to maximize certain desirable properties of the coating or "the smaller the better" - used when it was expedient to minimize certain undesirable propertiesof the coating. The variation intervals used of the selected deposition parameters under optimization are provided in Table 2.

Table 2.
Optimized deposition parameters of DLC coatings

Optimized parameter	Variability interval		
	1	2	3
Substrate bias voltage U_B (V)	floating	-20	-80
Argon pressure p_{Ar} (Pa)	0.01	0.05	0.25
DLC coating thickness t_{DLC} (µm)	0.9	1.4	1.8
Cr sublayer thickness t_{Cr} (µm)	0.03	0.1	0.3

Table 3 includes a list of the deposition parameters under optimization, i.e. substrate bias voltage U_B, argon pressure p_{Ar}, thickness of DLC coating t_{DLC}, thickness of Cr sublayer t_{Cr} as well as the denotations of the experiments.

Table 3.
Table of experiments

Exp. No.	Substrate bias voltage U_B (V)	Argon pressure p_{Ar} (Pa)	Thickness of DLC coating t_{DLC} (µm)	Thickness of Cr sublayer t_{Cr} (µm)
1	floating	0.01	0.9	0.03
2	floating	0.05	1.4	0.1
3	floating	0.25	1.8	0.3
4	-20	0.01	1.4	0.3
5	-20	0.05	1.8	0.03
6	-20	0.25	0.9	0.1
7	-80	0.01	1.8	0.1
8	-80	0.05	0.9	0.3
9	-80	0.25	1.4	0.03

2.4. Investigation results and discussion

In the initial phase, a number of the properties of the DLC coatings were verified: the chemical composition, adhesion, hardness, the modulus of elasticity, the roughness of the surface as well as the wear rates of the coatings and counter-samples. After an analysis of the results obtained, the adhesion (L_c) of the coatings was recognized to be an important property from the perspective of a modification of the surfaces to woodworking tools. Critical load L_c, hardness H and the wear rate of the coating k_{vA} in frictional contact with Al_2O_3 ceramics were accepted as the optimization criteria.

The DLC coatings under examinations are characterized by an amorphous and glass-like compact structure without any fractures or delamination. On the coating surface, there occurs a droplet phase which is characteristic of the arc method (Fig. 1). A divergence of thicknesses of ca. 10% of the values obtained from the values accepted in the experiment table (Table 2) was established on the basis of an analysis of the measurement results of the thicknesses of DLC coatings that were determined by means of the profilographometric method and on the basis of SEM observations. The analyses of the chemical composition with the use of the EDS method indicate that DLC coatings contain ca. 95-97% at. of carbon and 3-5% at. of oxygen, whereas the argon contents are negligible on the measuring method error level.

The values of L_{c2} critical load at which there occur distinct and cyclically appearing damages (cracks and losses) of the DLC coating, which are established on the basis of observations with an optical microscope, were accepted as the basic values for the purpose of the determination of the critical load which causes an adhesive damage of those coatings that were deposited on substrates from HS6-5-2 high-speed steel. Due to the fact that initiating (single) damages to the coating with L_{c1} load do not always occur and due to the difficult determination of an unequivocal value of L_{c3} at which there occurs a total removal of the coating, very frequently, after a complete rupture of the coating at higher loads, the coating reoccurs periodically on the whole width of the scratch track (Fig. 2).

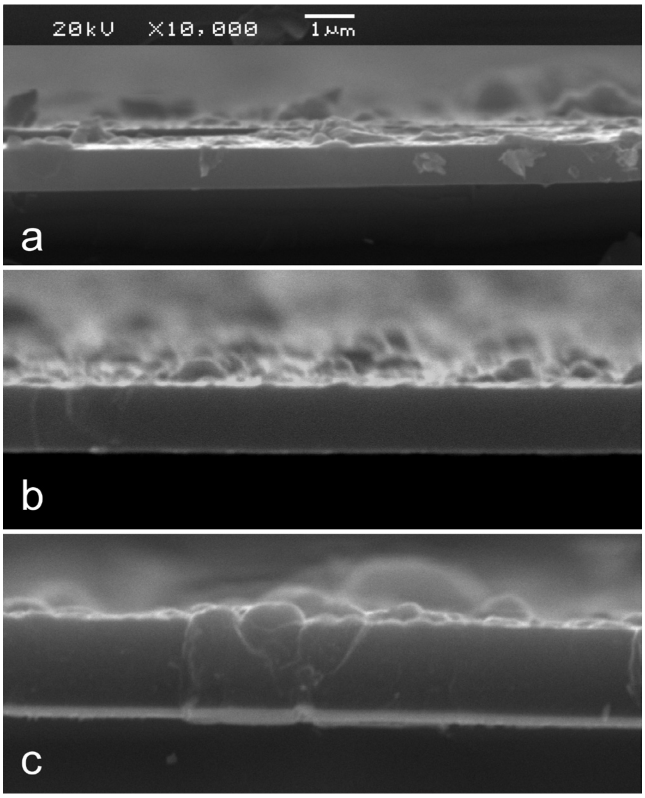


Fig. 1. SEM fracture images of DLC coatings with a Cr sublayer: a) DLC - 0.9 μm, Cr - 0.03 μm; b) DLC - 1.4 μm, Cr - 0.1 μm; c) DLC - 1.8 μm, Cr - 0.3 μm

The results of the examinations of the adhesion with the scratch method (L_{c2}), hardness (H) and wear rate (k_{vA}) of the DLC coatings that were deposited in successive experiments on an HS6-5-2 steel substrates are presented in Table 4.

All DLC coatings exhibit adhesion, specific critical load L_{c2} in the range of 19-35 N (Table 4). The results of a statistical analysis of the influence of the technological parameters optimized on critical load L_{c2} are presented in Fig. 3. A high argon pressure of 0.25 Pa, the floating potential of the substrates and the application of a metallic Cr sublayer of the thickness of 0.3 μm have a favorable effect on the adhesion of DLC coatings to high-speed steel substrates (cf. Fig. 3). A similar positive influence of metallic sublayers on the adhesion of carbon coatings through a reduction of stresses between the hard coating and the substrate was also established by [4,9,15].

The results of adhesion tests with the use of the Rockwell method, which are presented in Fig. 4, confirm that DLC coatings are characterized by a good adhesion of HF1-HF2 to HS6-5-2 steel substrates when a high argon pressure (0.25 Pa) and a high Cr sublayer (0.3 μm) were used. The cracks and losses (HF3-HF4) of the coating, which prove poor adhesion, occur for those DLC coatings that were deposited with a low argon pressure (0.01 and 0.05 Pa) with a Cr sublayer of a small thickness (0.03 and 0.1 μm).

The hardness of DLC coatings changes in the range of 20-66 GPa (cf. Table 4). The results of a statistical analysis of the influence of the deposition parameters optimized on hardness are presented in Fig. 5. A high energy of ions which is obtained with high values of the substrate bias voltage (U_B = -80 V) and a low argon pressure (p_{Ar} = 0.01 Pa) exert the greatest influence on an increase of the hardness of DLC coatings. Similar dependencies were described in the papers [10,14,15,19].

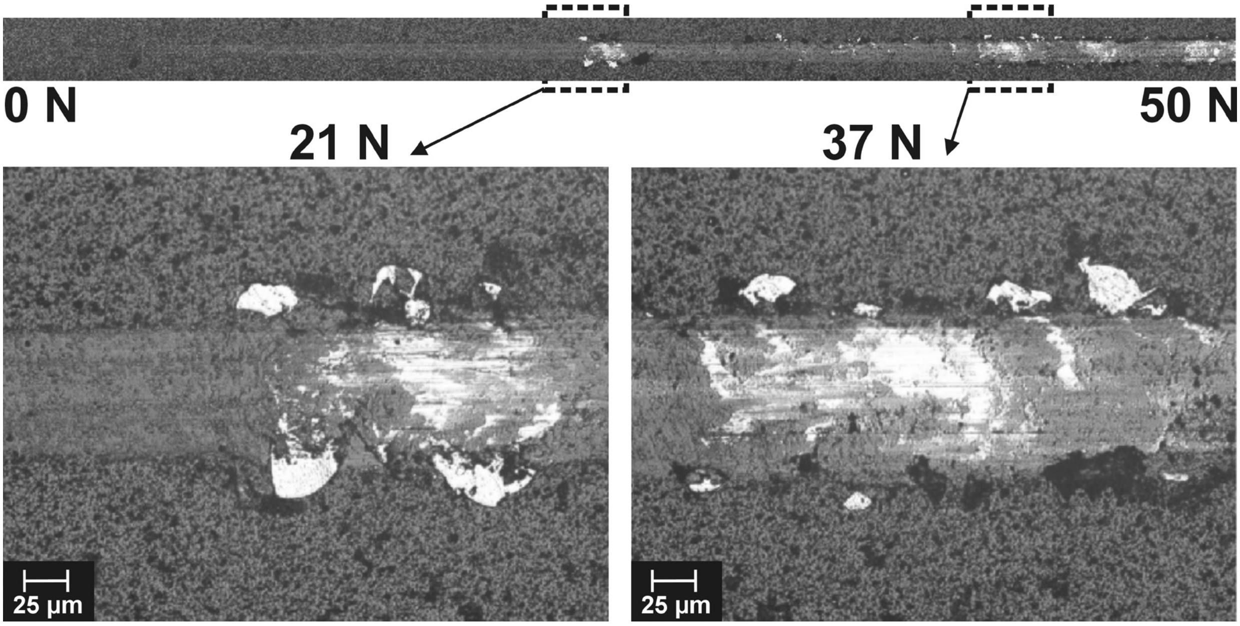


Fig. 2. Images of characteristic damages after a scratch test of the DLC coating that was deposited on an HS6-5-2 steel substrate

Table 4.
Values of critical load L_{c2}, hardness H and wear rates k_{vA} of DLC coatings deposited on an HS6-5-2 steel substrates

Exp. No.	Adhesion L_{c2} (N)			Hardness H (GPa)			Wear rate k_{vA} $\times 10^{-7}$ (mm^3/Nm)		
	I	II	III	I	II	III	I	II	III
1	22	22	21	32.6	34.4	37.0	1.9	2.4	3.7
2	26	26	22	29.3	32.0	33.6	2.4	3.9	5.7
3	33	35	31	20.3	23.1	35.3	4.0	4.4	6.0
4	22	19	21	49.4	54.1	66.2	2.0	2.1	4.5
5	25	22	22	33.5	37.3	43.8	3.6	4.6	5.4
6	22	20	23	32.5	33.7	35.6	3.2	3.8	4.3
7	23	21	22	57.2	61.2	64.2	1.7	5.2	6.9
8	22	22	22	34.8	44.2	63.1	1.2	2.1	3.1
9	24	22	22	40.1	41.6	42.2	3.6	3.9	4.1

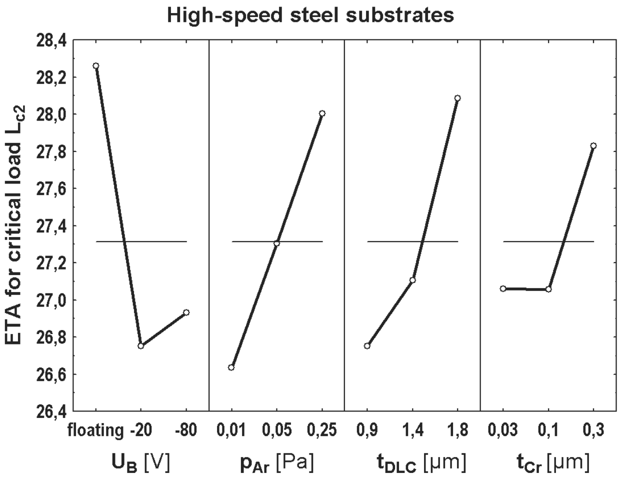
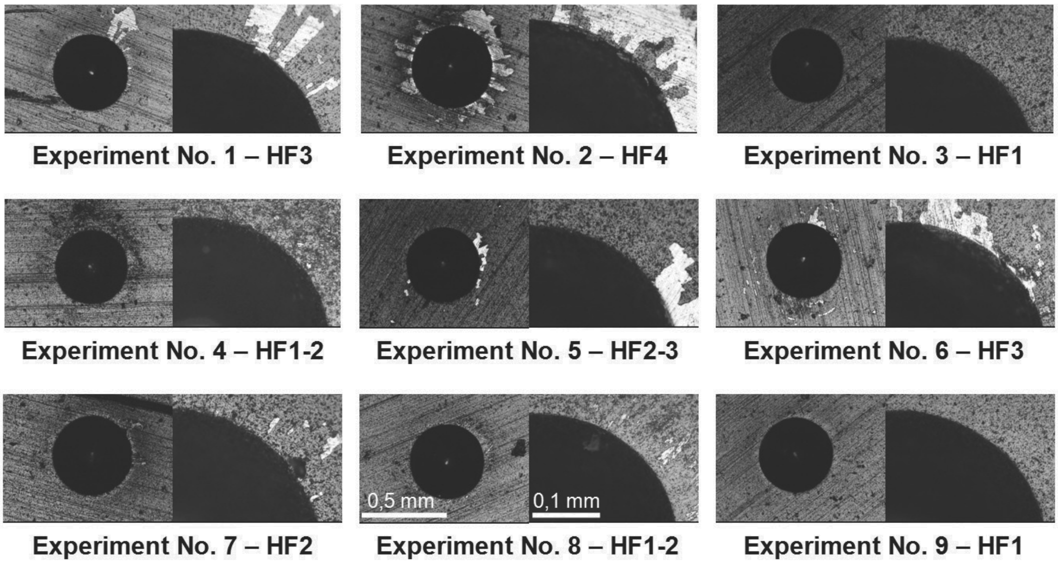


Fig. 3. Results of a statistic analysis of the impact of the deposition parameters on critical load L_{c2} of DLC coatings that were deposited on an HS6-5-2 steel substrates



Experiment No. 1 – HF3
Experiment No. 2 – HF4
Experiment No. 3 – HF1
Experiment No. 4 – HF1-2
Experiment No. 5 – HF2-3
Experiment No. 6 – HF3
Experiment No. 7 – HF2
Experiment No. 8 – HF1-2
Experiment No. 9 – HF1

Fig. 4. Characteristics of the adhesion of DLC coatings that were deposited on an HS6-5-2 steel substrates, determined in Rockwell test

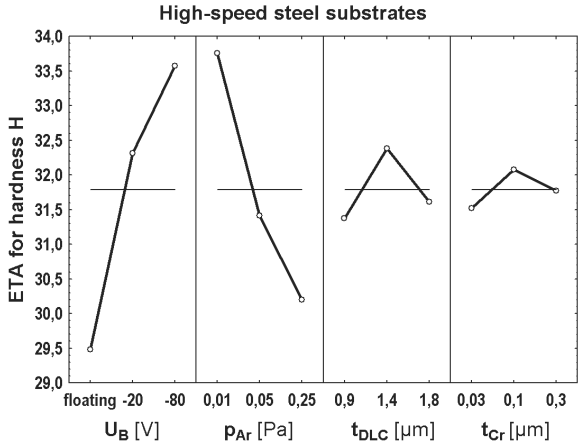


Fig. 5. Results of a statistic analysis of the impact of the deposition parameters on hardness H of DLC coatings that were deposited on an HS6-5-2 steel substrates

The results of a statistical analysis of the influence of the deposition parameters optimized on the wear rate kvA are presented in Fig. 6. Wear resistant DLC coatings i.e. those with low values of wear rate kvA in a frictional contact with Al2O3 ceramics were deposited at a high value of the substrate bias voltage (UB = -80 V) and a low argon pressure (pAr = 0.01 Pa). A high wear resistance may result from a high hardness of those DLC coatings that were deposited with such technological parameters as well as from the possibility of a graphitization of carbon coatings, which was described by [10,17,19].

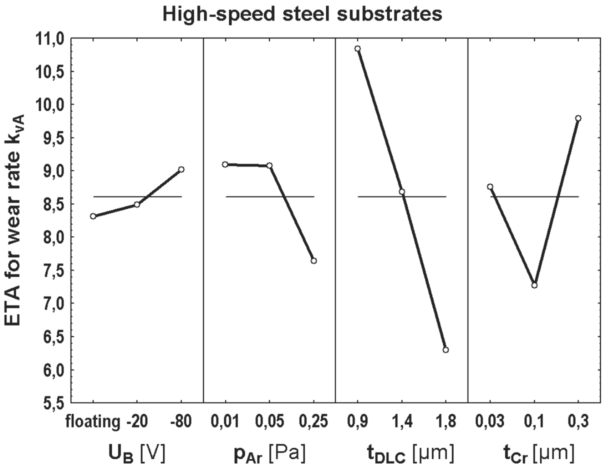


Fig. 6. Results of a statistic analysis of the impact of the deposition parameters on wear rate k_{vA} of DLC coatings that were deposited on an HS6-5-2 steel substrates

2.5. Verifying experiments

On the basis of the results obtained of the examinations of DLC coatings and statistical analyses in compliance with the experiment planning method according to Taguchi, verifying experiments were conducted with the use of optimal parameters which make it possible to obtain the following:

- a maximum adhesion on an HS6-5-2 steel substrates: W-L_{c2},
- a maximum hardness of coatings: W-H,
- a minimum wear rate (k_{vA}) in frictional contact with ceramics Al_2O_3: W-k_{vA}.

The optimal deposition parameters and the results of the investigations into the properties of those DLC coatings that were deposited in verifying experiments on an HS6-5-2 steel substrates are presented in Table 6.

Table 5 includes the optimal values of the deposition parameters of DLC coatings on an HS6-5-2 steel substrates that were determined on the basis of statistical analyses (cf. Figs. 3, 5, 6) for the purpose of obtaining their maximum adhesion (L_{c2}), hardness (H) and the minimum wear rate (k_{vA}).

Fig. 7 includes the images of characteristic damages after a scratch test of the DLC coating which was deposited in a verifying experiment (W-L_{c2}) on an HS6-5-2 steel substrates. The average value of critical load L_{c2} was ca. 30 N (cf. Table 6) and was close to the highest values that were obtained in the previous experiments (cf. Table 4).

The average hardness of DLC coatings of ca. 57 GPa, which was obtained in a verifying experiment (cf. Table 6), was nearly to the higher average values that were obtained in the previous experiments (cf. Table 4).

The wear rate ($k_{vA} = 3.7\times10^{-7}$ mm^3/Nm) of the DLC coating which was deposited in a verifying experiment W-k_{vA} (Table 6) was slightly higher than the lowest ones which were obtained in the previous experiments (cf. Table 4).

3. Summary and conclusions

The optimization method used permits obtaining a lot of important information related to the influence of the deposition parameters on the properties of DLC coatings with a relatively small number of experiments.

The thickness of the DLC coating and of the Cr sublayer has a significant influence on the adhesion of coatings. In order to ensure a high adhesion to HS6-5-2 high-speed steel substrates, a thick Cr sublayer (0.3 µm) and DLC coating (1.8 µm) that is deposited at a high argon pressure p_{Ar} (0.25 Pa) are to be used; no substrate bias (a floating potential) is to be used.

The value of the substrate bias voltage and the argon pressure exert the greatest influence on the hardness and frictional wear of DLC coatings. In order to obtain a high hardness H and wear resistance that is determined with wear rate k_{vA}, higher values of substrate bias voltage U_B (-80 V) and low argon pressure p_{Ar} (0.01 Pa) are to be used. Depending from the deposition parameters used, it is possible to obtain DLC coatings in a wide hardness interval of 20-60 GPa.

The properties of those DLC coatings that are deposited with optimal parameters as well as the information from the studies by [4,5,9,11] may indicate the possibility of their use on woodworking tools or tools for wood-like materials for the purpose of increasing their durability.

Table 5.
Optimal values of deposition parameters to obtain a maximum adhesion, hardness and a minimum wear rate of DLC coatings which are deposited on an HS6-5-2 steel substrates

Evaluation criteria of coatings	Optimal values of deposition parameters			
	U_B (V)	p_{Ar} (Pa)	t_{DLC} (μm)	t_{Cr} (μm)
Adhesion L_{c2}	floating	0.25	1.8	0.3
Hardness H	-80	0.01	1.4	0.1
Wear rate k_{vA}	-80	0.01	0.9	0.3

Table 6.
Results of investigations into the properties of DLC coatings that were deposited in verification experiments on an HS6-5-2 steel substrates

Exp. denotation	Optimal parameters for L_{c2}				Optimization criteria		
W-L_{c2}	U_B (V)	p_{Ar} (Pa)	t_{DLC} (μm)	t_{Cr} (μm)	L_{c2} (N)	H (GPa)	k_{vA} ×10^{-7} (mm^3/Nm)
	floating	0.25	1.8	0.3	30	20.5	6.2
W-H	Optimal parameters for H				Optimization criteria		
	U_B (V)	p_{Ar} (Pa)	t_{DLC} (μm)	t_{Cr} (μm)	H (GPa)	L_{c2} (N)	k_{vA} ×10^{-7} (mm^3/Nm)
	-80	0.01	1.4	0.1	57.1	19	3.0
W-k_{vA}	Optimal parameters for k_{vA}				Optimization criteria		
	U_B (V)	p_{Ar} (Pa)	t_{DLC} (μm)	t_{Cr} (μm)	k_{vA} ×10^{-7} (mm^3/Nm)	L_{c2} (N)	H (GPa)
	-80	0.01	0.9	0.3	3.7	24	32.1

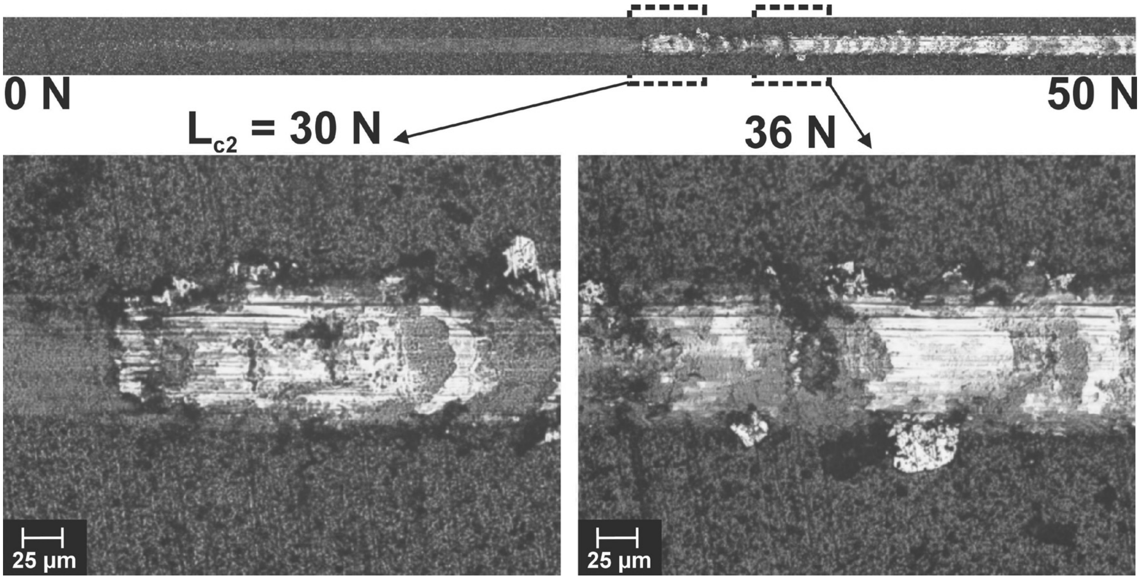


Fig. 7. Images of characteristic damages after a scratch test of the DLC coating that was deposited on an HS6-5-2 steel substrates in verification experiment W-L_{c2}

Acknowledgements

This work was supported by the Operational Programme Innovative Economy POIG 2007-2013 within Developmental Project No. UDA-POIG.01.03.01-32-052/08-00: "Hybrid technologies for woodworking tools modification".

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