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Adhesion of PVD and CVD hard coatings as an essential parameter that determines the durability of coated tools

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ABSTRACT

Purpose: The work is connected with the current trend related to the modification of tool surfaces with PVD and CVD methods through the deposition of coatings to increase their durability. The research results of coated tools tests that are carried out in industrial conditions are presented in details.

Design/methodology/approach: Structure, chemical and phase composition investigations related to the mechanical and tribological properties of coatings produced on tool substrates and analysis of the results are included. Investigations of the properties of deposited coatings on the following tool materials were made: high speed steels, hot work tool steel, sintered carbides and SiAlON tool ceramics.

Findings: Interpretation of production tests results of coated tools and an analysis of the wear mechanisms of tool blades in relation to the properties of coatings and their adhesion, in particular characterized in the scratch test, were described.

Research limitations/implications: Adhesion scratch test cannot be the only and final method of such evaluation. For example a direct comparison of the results of the scratch tests of coatings is possible when adhesion is being examined of different coatings yet on the same type of the substrate.

Practical implications: Based on the adhesion test results using the scratch test, the suitability of the coatings produced on the cutting tools can be quickly assessed.

Originality/value: It was sought those parameters which characterize the properties of coatings, ones which would permit to effectively/practically assess the performance of tools with coatings produced, with an exclusion or limitation of long-term and expensive durability tests of tools that are carried out in industrial conditions.

Keywords: Hard coatings, Tool materials, Adhesion, Scratch test, Production tests, Tool lifetime, Wear mechanism

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MATERIALS

1. Introduction

In connection with continuously growing requirements in relation to cutting tools concerning an increase of the machining speed, the necessity of the working of hard materials and hard machinable materials or use in machining centres, there occurs a need to increase their durability and reliability. To meet these requirements, new tool materials are developed, the properties of the materials used so far are modified through heat treatment or thermochemical treatment, changes are made in the geometrical parameters of tool blades and the appropriate cooling lubricants are selected. In the past several dozen years, there has been a significant progress in the surface engineering of tool materials owing to the development of the deposition technology of coatings with CVD (Chemical Vapour Deposition) and PVD (Physical Vapour Deposition) methods on the tool surface. The use of single-layer or single-phase coatings no longer ensures sufficiently the requirements connected with the durability and reliability of tools. For this reason, various technologies are currently being developed to produce multi-layered, gradient or nanostructured coatings.

The cost of cutting tools constitutes 2-5% of the total machining costs in the production process of details. An extension of the life of tools alone has no greater influence on the total manufacturing cost. An increase of the durability of tools by 50% causes a reduction of the manufacturing costs of an article by merely 1%. However, a fundamental portion of the costs can be reduced because coated tools may work with a larger productivity. This leads to a reduction of the machine costs of production. Greater machining speed and a larger thickness of the layer machined significantly increase the production efficiency. The possibility to increase the speed machining by 20% may cause a reduction of costs by 15% per one workpiece [1,2]. By using more efficient and durable tools, one may also limit stoppages caused by the changes of tools as well as limit or eliminate cooling lubricants. The innovative methods of the modifications of tool surfaces also permit an effective machining of workpieces from hard machinable materials.

To be effective, anti-wear coatings produced on tool blades should possess a number of adequately selected parameters, including the chemical and phase composition, structure, thickness, surface smoothness, hardness, low friction coefficient and good adhesion to the tool material. Mechanical methods used in the characterization of the adhesion of coatings allow one to stimulate pressures and stresses which are similar to the real operating conditions

of coating – substrate systems, and the strength and energy under which there occurs a complete or partial separation of the coating from the substrate is defined as the experimental measuring result [3-5]. From among mechanical quantitative methods that characterize the adhesion of coatings, the scratch test is the most adequate for the investigation of the adhesion of coatings produced on cutting tools. During the test the sample moves linearly and indenter is pushed into the coating with increasing normal load. The indenter most frequently is in the form of a Rockwell diamond cone with the angle of 120° and tip rounding of 0.2 mm. The normal force which permits the appropriate value of mechanical force or energy required for adhesive breaking the coating off the substrate is defined as the critical load L_C . During the determination of the critical load in the scratch test, significant unit pressures which are similar to those that occur on the tool blade during machining occur.

Models and their modifications concerning the adhesive destruction mechanisms of the coating – substrate system in the scratch test are presented in the literature. Benjamin and Waver [6] describe the critical shear stress in the coating-substrate transition layer as the function of the applied load, substrate hardness and the tip radius of the indenter. In Laugier [7] assumes that during the scratch test, there occurs mainly an elastic strain of the substrate, and the presence of adequately high compressive stresses in the coating in the area before the moving indenter is essential for the coating being broken off from the substrate. In the model based on the elastic-plastic indent theory, using the notion of an interfacial constraint of the coating and the substrate, Burnett and Rickerby [8] demonstrated that knowledge of the stress and deformation state makes it possible to calculate accumulated energy, which may be of an essential importance for breaking the coating off. Based on the described models of wear [6-8] being the result of the scratch test, and based on the observations and identification of damage to the coating, the value can be determined of the normal force causing cohesive damage in the coating as well as damage that proves that the adhesion forces between the coating and the substrate have been broken off. The scratch test is usually carried out with automated devices that allow an adjustment of the increment speed of the normal load and the displacement speed of specimen with the coating deposited [4,8]. In order to determine the value of the critical load L_C showing that adhesion forces have been exceeded, the frictional force and the acoustic emission are measured, or microscopic observations are performed to identify the characteristic defects of the coating. As it is not always that the coating is completely removed from the

scratch trace, special map-patterns of defects that take into account the impact of the substrate hardness and the coating hardness on the nature of the occurring cohesion and/or adhesion defects of coatings are presented in the literature [8-10].

The work is connected with the current trend related to the modification of tool surfaces with PVD and CVD methods through the deposition of coatings to increase their durability. By using many modern research methods, it was determined the chemical and phase composition as well as the mechanical and tribological properties of coatings. It was sought those parameters which characterize the properties of coatings, ones which would permit to effectively/practically assess the performance of tools with coatings produced, with an exclusion or limitation of long-term and costly durability tests of tools that are carried out in production conditions.

Table 1.
Type of characterized coatings and tool substrates

Type of coating	Tool substrate	Reference
Single-layered		
TiN, TiCN, TiC, DLC	High speed steel	[11-13, 18-21]
CrN	Hot work tool steel	[17]
Multi-layered		
Ti(C,N)+TiN (CVD)	SiAlON tool ceramics, sintered carbides WC-Co	[15]
Cr/CrN/Cr/DLC,	High speed steel, sintered carbides WC-Co	[20]
Cr(CrN/CrCN)/Cr/DLC	High speed steel	[21]
Gradient		
Ti(C,N), (Ti,Zr)N, (Ti,Al)N, (Al,Ti)N, (Al,Cr)N	SiAlON tool ceramics, sintered carbides WC-Co	[14-16]
Nanostructured		
CrAlSiN, CrAlSiN+DLC, WC/a-C:H	Hot work tool steel	[17]
W-DLC	High speed steel, sintered carbides WC-Co	[20]

The adhesion of the coatings to the substrates was characterized by a CSM Instruments S.A. Revetest® Scratch-Tester. The C Rockwell indenter (radius 200 μm) type was used. 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 (0-50 N or 0-200 N); normal loading rate 100 N/min (or 200 N/min); relative sliding speed of the table with the sample 10 mm/min; the scratch length 10 mm or 5 mm; distance between successive scratches ca. 1 mm [11-21]. 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.

2. Substrates, coatings and adhesion test methodology

The paper describes the deposition technology of hard coatings using PVD and CVD methods, as well as the analysis of the properties of tool substrates and coatings deposited on various tool materials, presented in previous works of the author [11-21]. Investigations of the properties of tool substrates and deposited coatings on the following tool materials were made: high speed steels, hot work tool steel, sintered carbides, tool ceramics. Type of coatings and tool substrates, characterised in this work, are presented in Table 1. Parameters of coating deposition and detailed description of the methodology of research on structure, chemical composition and properties of coatings had been described in earlier works of the author [11-21].

With L_{C2} critical load, there occur distinct and regular damages to the coating; with L_{C3} critical load, there usually occurs a total delamination of the coating [8-10].

3. Results and discussion

The studies [11-13] were related to the modification of the surface of high speed steel drills, through the production of anti-wear coatings with the PVD technology, which is in line with the current trend in the tool industry. High speed steel tools constitute at present ca. 20% of the global market of machining tools, and drilling processes constitute ca. 17% of all machining methods [2]. In the studies [11-13] it was presented research results related to the properties of titanium carbon nitride coatings produced

with the cathodic arc plasma deposition, in the mixture of the reactive gases of nitrogen and acetylene. The coatings were deposited on specimens [11,12] as well as on tools and test specimens from HS6-5-2 high speed steel [13].

The chemical and phase composition of coatings produced with changes in the share of nitrogen in the mixture of reactive gases in the range of 0-100% are described in the study [11]. It was conducted X-ray diffraction structural tests and an analysis of the chemical composition of the coatings using the following methods: EDS, GDOES, RBS and ERDA. The X-ray structural tests and the analyses of the chemical composition of the coatings show that there occurs a solution of solid carbon and nitrogen in Ti(C,N) with a chemical composition and a lattice parameter contained between titanium nitride and titanium carbide. In the tests of the chemical composition (GDOS, RBS), it was confirmed the occurrence of a transition zone of a diffusive nature between the coatings and the steel substrate. SEM tests of fractures show that all the coatings possess compact and fine grain structure, and they adhere well to high speed steel substrates owing to the use of the Ti+TiN sublayer with metallic bonds with the substrate, which increase their adhesion. The results of tribological tests conducted in dry friction conditions with the ball-on-disk method of titanium carbon nitride were presented in the study [12]. In order to determine the abrasive wear of coatings and substrates from HS6-5-2 steel, frictional couples with ceramic Al_2O_3 and Si_3N_4 counterspecimens were used. The counterspecimens from 100Cr6 bearing steel and EN-GJL-250 grey cast iron, which belong to the group of iron casts, as materials that represent/simulate potential materials processed were applied. It was determined the dry friction coefficient for all frictional couples and calculated the wear rate of the coatings and counterspecimen. It was conducted an analysis of the wear mechanisms of the coatings and counterspecimens based on SEM microscope observations, 3D profilograms of the traces of abrasion and EDS linear profiles of the chemical composition of coatings. The highest values of the wear rate of the coatings occurred for the frictional contact with ceramic counterspecimens. The wear of the coatings and the wear of the counterspecimens increases for coatings that are produced with a high share of nitrogen; this is connected with an increase of the coefficient of friction. For TiC and Ti(C,N) coatings with low nitrogen concentration, the lowest values of the dry friction coefficient as well as the wear rates of the coatings and counterspecimens were measured. Based on the tribological properties and analyses of the chemical composition [11] found that those coatings that are specified as titanium carbide are Ti-C:H type coatings,

which are characterized by a low coefficient of friction. In the friction pair there are complex physical and chemical processes of mutual wear. Adhesive wear, related to the displacement of the material in the friction pair, occurred between titanium carbon nitride coatings with a high nitrogen concentration and TiN coatings and counter-specimens from 100Cr6 steel and EN-GJL-250 cast iron. It was demonstrated that, apart from frictional and adhesive wear, there also occurs oxidation, which indicates chemical wear in the frictional contact area. The most intensive oxidation of the coatings occurred when using ceramic counterspecimens [12].

Based on an analysis of the research results obtained and described in the studies [11,12], it was established the deposition parameters of coatings on twist drills from HS 6-5-2 high speed steel. A Ti+TiN sublayer was used to improve the adhesion of titanium carbon nitride and titanium carbide coatings. The coatings selected for industrial tests exhibited a significant diversification of properties, for example: the highest adhesion, characterized in the scratch test by the critical load $L_C = 86$ N, was exhibited by TiN titanium nitride. Titanium carbon nitride Ti(C,N) possessed the highest hardness of ca. 32.7 GPa and titanium carbide had the lowest dry friction coefficient of 0.18 and lowest wear rate in frictional couple with cast iron [12, 13]. It was used the uncoated and coated tools tests of dry drilling, castings of brake disks made from EN-GJL-250 grey cast iron. It was described these research results in the study [13]. Based on the measurements of the limit values of those indices that characterize the wear of the drills, it was found out that tools with a titanium carbon nitride and titanium nitride coating possess almost a two-fold higher durability related to uncoated tools and tools with a TiC coating (Fig. 1). Due to the increased temperature in the cutting area and a great affinity of carbon to iron from the material processed, there might have occurred intensive chemical wear of TiC coatings. TiC coatings, which showed in tribological tests [12,13] the lowest coefficient of friction and the lowest wear rate, were subject to intensive wear from the working surfaces of the drill blade; what was left was only the adhesive T+TiN sublayer, which may be the result of the four-fold higher sliding speed that occurs during machining in relation to the speed used during tribological tests. By increasing the durability of the drills, it was possible to limit the downtime related to the change of tools in the multi-spindle machine tool. Based on SEM observations and an analysis of the chemical composition with the EDS method, it was established that there is a built-up of the material machined on the blades of uncoated tools and on the worn edges of coated blades. This may indicate that there is a large

contribution of the adhesion mechanism of the wear of the tools (Fig. 1). The forms of the wear of the blades show that TiN and Ti(C,N) coatings limit abrasive wear and the tendency of the production of built-up [13].

It was demonstrated that through changes of the share of nitrogen in the atmosphere of reactive gases, it is possible to produce on high speed steel, with the cathode arc evaporation method, Ti(C,N) coatings with a significant diversification of mechanical and tribological properties [11-13]. Ti(C,N) coatings deposited with a high share of nitrogen, demonstrated the highest hardness, a low coefficient of friction in relation to cast iron, and high

adhesion to high speed tool steel owing to the use of the Ti+TiN sublayer. Owing to these properties, there was a two-fold increase in the durability of these drills through a reduction of abrasive wear and a reduction of the tendency of the built-up of the material processed on the surfaces of the blades of the tools modified in this manner. The analysis conducted of the properties of TiN and Ti(C,N) coatings as well as of the wear mechanisms of the blades of the tools coated after production tests demonstrates a close link between the high adhesion of these coatings, determined by the critical load L_C in the scratch test and their increased durability.

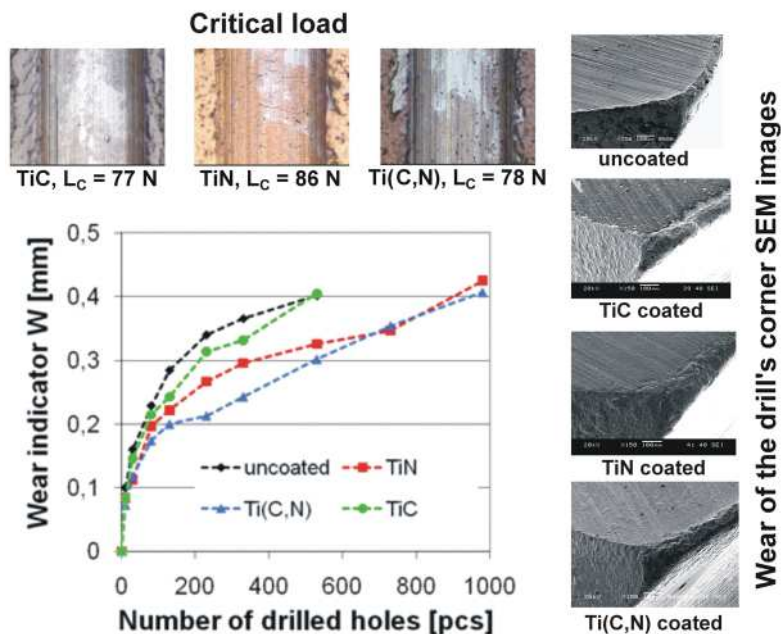


Fig. 1. Wear of the drill's outside corner (W indicator) in the function of the number of drilled holes, SEM image of the drill's corner wearing and images of coating damage at critical load L_C in the scratch test. Based on [11-13]

The issues discussed in the studies [14-16] concerned modifications of the surfaces of removable indexable inserts made from sintered carbides WC-Co and from SiAlON tool ceramics, through the production of gradient coatings with the PVD technology [14-16] and multilayer coatings with the CVD technology [15]. Hard and super-hard tool materials are most frequently used in the form of removable indexable inserts, and they are mainly used for the machining of cast iron, alloys based on nickel and hardened steels. Until recently, the belief was widespread that deposition of coatings on cutting blades is not justifiable due to its high hardness. The use of hard coatings with a lower coefficient of friction may reduce the amount of heat produced in the cutting process as a result

of friction, and thereby it may increase the durability of the tools coated. In the global market of cutting tools from hard and super-hard materials, sintered carbides currently possess a 53% market share with 10% market share for tool ceramics. Every year, there is an increase in the sale of tools from hard and super-hard materials by 4% to 5%. The sale of tools from sintered carbides without any coatings is decreasing in time. The share of sintered carbides with coatings is growing on a large scale. The greatest increase in sale is observed for sintered carbides with coatings produced using the technologies of PVD [1,2]. In the global market of all sintered carbides in the year 2003, it was only 22% that were covered with the use of PVD technologies, whereas in the year 2013, sintered carbides

with coatings produced using PVD technologies constituted as much as 60% of the total market of carbides. Material removal processing by turning constitutes ca. 30% of all the cutting methods used [2].

The adhesion of Ti(C,N) and (Ti,Zr)N gradient coatings was characterized in the scratch test through the determination of the value of the critical load L_C based on a sharp increase of the signal of acoustic emission AE,

an increase of the friction force F_t and based on the microscope observations of characteristic damages [14]. There was a significant conformity of the critical load value determined with the use of three detection methods. Both the gradient Ti(C,N) and (Ti,Zr)N coatings demonstrated high adhesion to sintered carbide substrates, which was characterized by the critical load L_C over 50 N (Fig. 2).

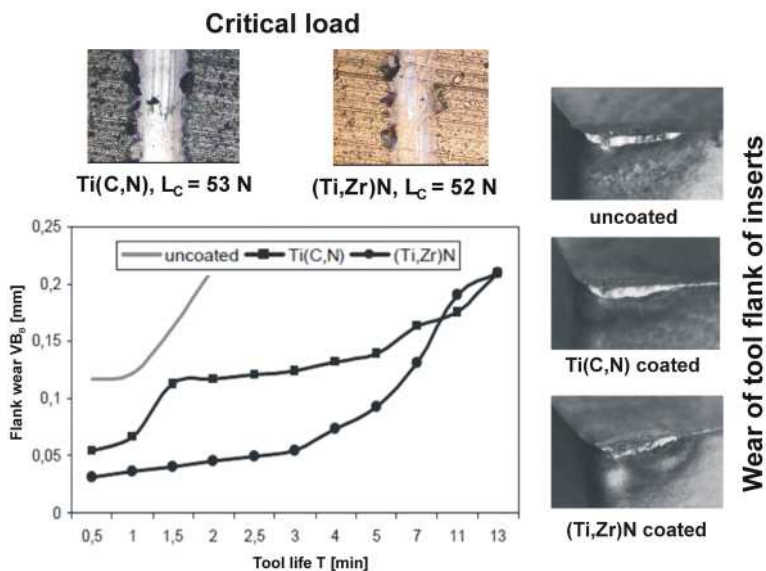


Fig. 2. Tool life of uncoated, Ti(C,N) and (Ti,Zr)N coated of sintered carbide inserts, images of wear of tool flank of inserts and images of coating damage at critical load L_C in the scratch test. Based on [14]

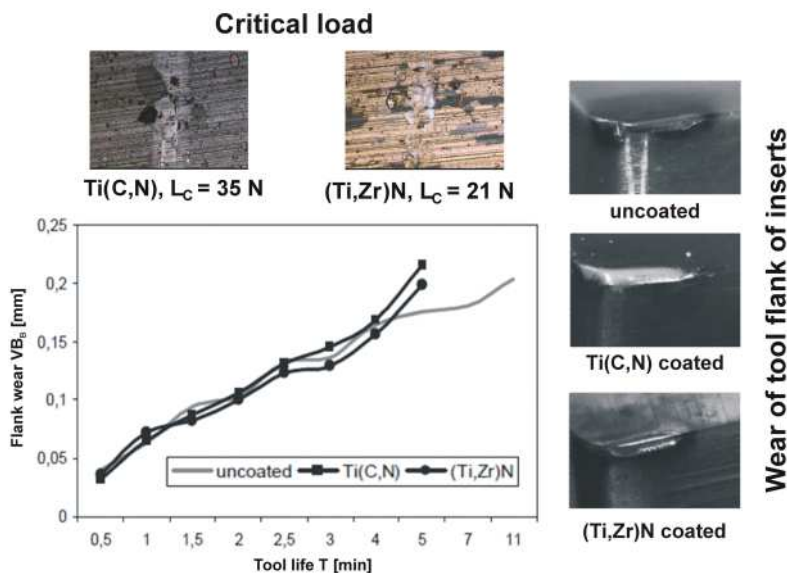


Fig. 3. Tool life of uncoated, Ti(C,N) and (Ti,Zr)N coated of SiAlON ceramic tool inserts, images of wear of tool flank of inserts and images of coating damage at critical load L_C in the scratch test. Based on [14]

The adhesion of the same coatings to SiAlON tool ceramics is substantially lower: for Ti(C,N), the critical load is $L_c = 35 \text{ N}$ and $L_c = 21 \text{ N}$ for (Ti,Zr)N respectively (Fig. 3). The wear resistance of removable indexable inserts with and without coatings was determined during technological cutting tests with dry turning of grey cast iron. The cutting time of the tools until reaching the critical value of the flank wear (0.2 mm) was constituted the durability criterion (Fig. 2 and Fig. 3). The high hardness and good adhesion of gradient coatings to sintered carbide blades results in a ca. six-fold increase of the durability of the tools. The same coatings produced on blades from SiAlON tool ceramics, in spite of their high hardness, do not increase the durability of the tools; quite the contrary, they even deteriorate it, which might be the result of their poor adhesion (Fig. 3).

Based on an analysis of the research results presented in the study [15], it was found that the mechanical properties of coatings, i.e. their hardness, depend on their structure. Ti(C,N) gradient coatings with their characteristic fine crystal structure, possess higher hardness compared to Ti(C,N)+TiN multilayer coatings that are built of crystallites with column structure. Both the type of the substrate used and the production technology have an impact on the adhesion of coatings to tool substrates. It was demonstrated that Ti(C,N)+TiN coatings formed by the CVD technology possess good adhesion both to substrates from sintered carbides and SiAlON ceramics (Fig. 4 and Fig. 5). With increasing of normal load, in the scratch test, there occur initially small cracks and abrasions of the Ti(C,N)+TiN coating, and with the critical load, there occurs its total adhesion breaking. The thin TiC diffusive-adhesive sublayer that is formed during the deposition of multilayered Ti(C,N)+TiN coatings with the CVD technology on substrates from sintered carbide WC-Co improves their adhesion, which is confirmed by the highest values of the critical load $L_c = 110 \text{ N}$ (Fig. 4). During the tests of the adhesion of Ti(C,N) gradient coatings, there occurred various mechanisms of damages depending on the substrates on which they were produced. On sintered carbide, initially there occurred small cracks and chippings on the edge of the scratch trace, and at the critical load, the coating was torn away along the entire width of the scratch (Fig. 4). In Ti(C,N) coatings deposited on the SiAlON substrate, even with small loads, there occurred cracks and chippings of the coating over the entire length of the scratch and on its edges, and at the critical load, there occurred its total breaking with vast peeling, which was a proof of their poor adhesion (Fig. 5).

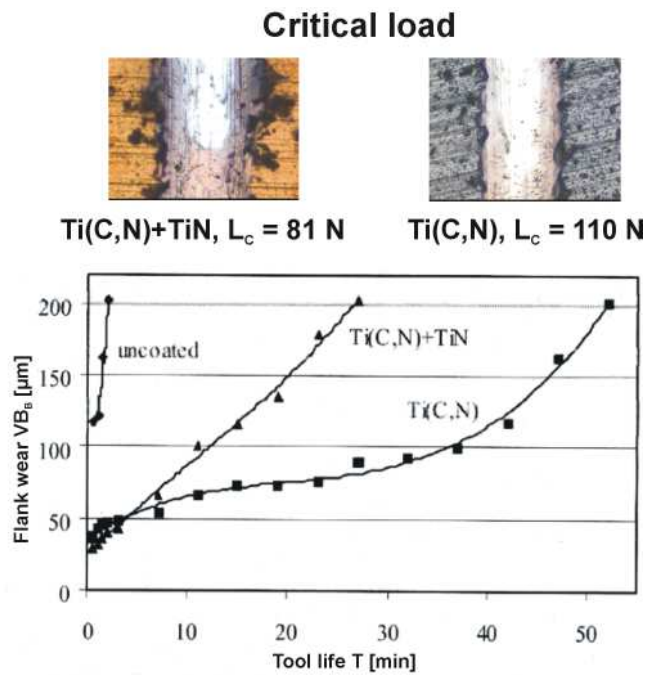


Fig. 4. Tool life of uncoated, Ti(C,N) and Ti(C,N)+TiN coated sintered carbide inserts and images of coating damage at critical load L_c in the scratch test. Based on [15]

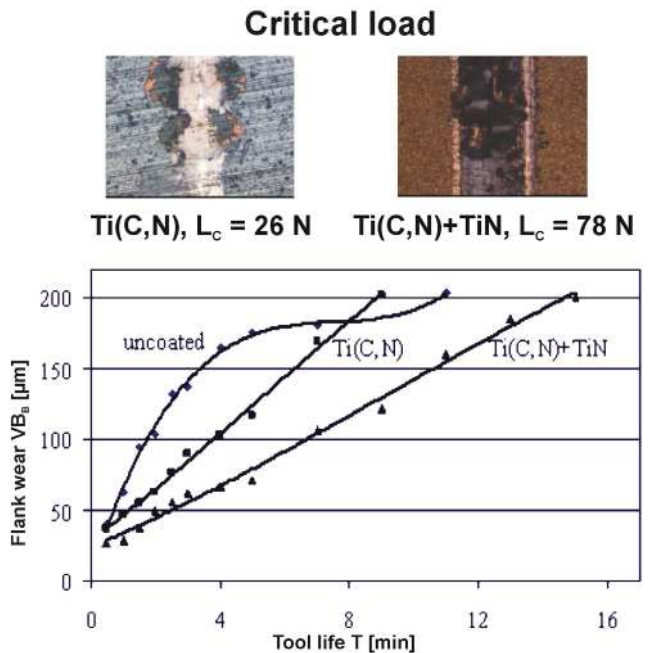


Fig. 5. Tool life of uncoated, Ti(C,N) and Ti(C,N)+TiN coated SiAlON ceramic tool and images of coating damage at critical load L_c in the scratch test. Based on [15]

The adhesion of Ti(C,N) and (Ti,Zr)N gradient coatings during the technological tests of cutting with dry turning of grey cast iron, the greatest durability of 52 minutes (Fig. 4) was exhibited by removable indexable inserts made from sintered carbides with a Ti(C,N) type coating produced with the PVD technology [15]. It is evident based on SEM microscope observations of worn cutting edges that it was abrasive wear that constituted the main cause of the wear of the blades. Removable indexable inserts made from SiAlON tool ceramics, with a Ti(C,N) coating, reached critical values of the wear of the blade as quickly as after 9 minutes of cutting, with visible vast peeling of the coating from the contact area. It is related to their poor adhesion and coating damage mechanisms, which are noticeable also once the critical load L_C has been reached in the scratch test (Fig. 5). SEM microscope observations and chemical composition analysis with the EDS method indicate the built-up of the material cut on the surface of the worn sintered carbide blade edges, which deteriorates cutting conditions [15].

Gradient coatings produced with the PVD technology, which are characterized in the study [16], present good adhesion to substrates from sintered carbides, which is the result of the adhesion and diffusion mechanism caused by a diffusion of elements in the coating-substrate transition zone, as a result of the implantation of high energy ions that are deposited on a negatively polarized substrate. The high values of the critical load $L_C = 112$ N, determined in the scratch test, indicate good adhesion of (Al,Ti)N coatings, deposited by PVD technology, to SiAlON tool ceramics (Fig. 6). This is caused by the presence of the AlN phase in the coatings with a hexagonal lattice, with covalent atom bonds of the same type as those in the ceramic substrate [16]. The mechanism of the damage of the coatings under examination depends mainly of their adhesion to the substrates, characterized in the scratch test. Those coatings which demonstrate the highest adhesion, characterized in the scratch test, high hardness and a fine grain structure, are subjected to damage mainly by abrasion, both during the scratch test and technological turning tests with cutting of grey cast iron (Fig. 6). Those coatings which demonstrate worse properties and, in particular, low values of the critical load L_C , which prove their poor adhesion, are subject to damage through cracking, peeling and delamination (Fig. 6).

Concluding the results obtained of the research into the properties of gradient and multilayer coatings produced on tools from sintered carbides and from tool ceramics [14-16]. The critical load L_C that is determined in the scratch test and which characterizes the adhesion of coatings to the substrate, depends to a significant extent

from the chemical and phase composition of the material of the coatings and tool substrates [14].

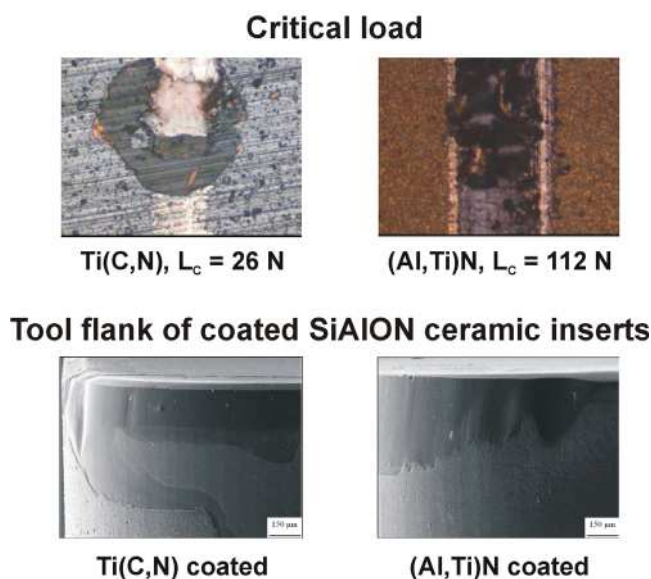


Fig. 6. Wear of tool flank of Ti(C,N) and (Al,Ti)N coated SiAlON ceramic inserts and images of coating damage at critical load L_C in the scratch test. Based on [16]

Gradient Ti(C,N) and (Ti,Zr)N coatings, where phases with metallic bonds of atoms dominate, have poor adhesion to SiAlON ceramics. This may be the result of the fact that in SiAlON ceramics covalent bonds dominate between atoms, from Si_3N_4 that is prevalent, as well as ionic bonds that are characteristic of Al_2O_3 . The structure of the coatings has a strong influence on the mechanical properties of tools coated [15]. The Ti(C,N) coatings with fine grain structure show better cutting ability as compared to Ti(C,N)+TiN coatings that are composed of larger crystallites. Those coatings that are characterized by high adhesion to sintered carbide WC-Co, high hardness and fine grain structure are subject to destruction by abrasion both during the scratch test and in a technological cutting test when grey cast iron is turned [15-16]. Those coatings that exhibit lower adhesion in the scratch test are more susceptible to cracking and substantial peeling and chipping from the working surfaces of the tool in the cutting test. The high hardness and good adhesion of the Ti(C,N)+TiN multilayer coating, which is produced on both substrates, as well as of the Ti(C,N) gradient coating, produced on sintered carbide substrates, have an influence on an increased tool-life of cutting tools [15]. Tools with these coatings may be used in cutting without the use of any cooling and lubricating liquids, which constitutes a very important environment friendly aspect. The type

of atom bonds that occur in the material of the tool substrate and in the coating is of a substantial significance to their adhesion [14-16]. This may constitute an important indication when selecting the coating material for cutting blades from tool ceramics and sintered carbides.

Tools and moulds for plastic working processes, die-casting or forming of non-ferrous metals are most frequently made from hot work tool steels. Due to the significant manufacturing costs of such tools, in order to increase their durability and to improve their technological properties, coatings are more and more frequently produced on their surfaces apart from heat treatment and nitriding. Owing to the use of PVD techniques, it is possible to produce anti-wear coatings below the tempering temperature during the heat treatment of such steels. I used the experience that I gained during the development of the deposition technologies [11-13] and an analysis of the properties of the coatings deposited on cutting tools [13-16] in the assessment of the properties of nanostructured coatings produced on X40CrMoV5-1 hot work tool steel, which is presented in the study [17]. It is evident based on SEM observations that CrAlSiN, CrAlSiN+DLC, CrN and WC/a-C:H coatings produced on X40CrMoV5-1 steel substrate showed a compact microstructure without delamination. The tests performed in a transmission electron microscope indicate that the coatings possess a nanostructured nature with small crystallites sized 3-13 nm [17,22]. The scratch test used in the assessment of the adhesion of nanostructured coatings shows that their damage begins with the value of the critical load L_{C1} , in the form of small damage of a cohesive nature and small adhesion damage (Fig. 7). The highest values of the load $L_{C1} = 22$ N were registered for CrAlSiN coatings. Characteristic defects that proved breaking of adhesive forces, of all the nanostructured coatings, occurred with the values of the critical load L_{C2} in the range of 45-55 N (Fig. 7), which confirms their good adhesion to substrates from X40CrMoV5-1 steel. The existence demonstrated in the study [17,22] of a transition zone between the steel substrate and the coating and between the individual components improves their adhesion.

The properties obtained of nanostructured coatings indicated their justifiable use in order to increase the wear resistance of dies from X40CrMoV5-1 steel, used in plastic moulding of an aluminium alloy, which was done by Lukaszewicz [22]. He accepted the condition of the surface of the product moulded and the wear of the face and of the calibrating belt of the tool as an assessment criterion of the service life of the die. In the case of dies with CrAlSiN+DLC layers produced on their surfaces, a threefold increase of their service lives was found as

compared to dies that are used as a standard in extrusion processes [22]. Considering the fact that even a small loss or damage of the anti-wear coating from the working surfaces of the die may disrupt the extrusion process and cause an insufficient quality of the product extruded to be obtained, the high adhesion of CrAlSiN+DLC coatings (Fig. 7), characterized in the scratch test [17,22], is an important property that contributes to the longest tool-life of coated dies [22]. An increase of the durability of dies, as a result of an effective work of the hard CrAlSiN nanostructured layer and the top low-friction DLC layer, is possible only until it preserves the required cohesion and appropriate adhesion to the steel die. The properties of the transition zone between the steel substrate and the coating as well as between the individual layers are of a great importance. Higher adhesion to the steel substrate results in a greater resistance to tribological wear.

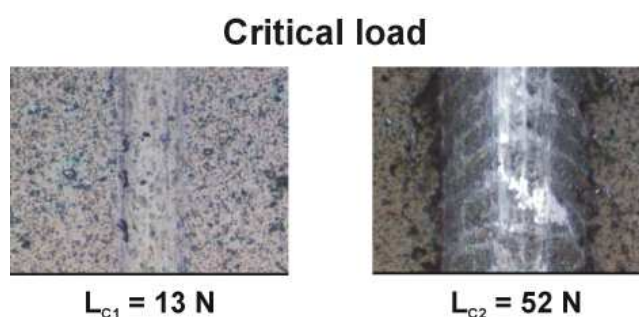


Fig. 7. Images of coating damage at critical load L_C in the scratch test of the CrAlSiN+DLC coating deposited onto the X40CrMoV5-1 tool steel substrate. Based on [17,22]

Comparing the properties of nanostructured coatings produced on hot working tool steel [17] and based on the results of operational tests are contained in the study [22]. The CrAlSiN+DLC coatings that consist of the internal nanostructured CrAlSiN layer that ensures greater hardness, strength and low thermal conductivity reduce the influence of internal factors on the wear process of dies for the extrusion of non-ferrous metals, while the external low-friction DLC layers provides good tribological properties. The proper formation of the transition zone between the material of the substrate and the CrAlSiN+DLC coating as well as between the individual layers in the coating, guaranteed an adequately high adhesion, which was characterized in the scratch test, and it made it possible to increase the tool-life of the coated dies.

The research issues referred to in the studies [18-21] were related to the development of the deposition technologies of anti-wear carbon based coatings (DLC)

on cutting tools for the processing of wood and wood based materials. As compared to metals, wood processing requires substantially higher parameters of cutting: the cutting speed is usually ten times as high, and the feed rate of the material ranges from several to a dozen or so m/min. With these processing parameters, tool edges undergo abrasive wear. This leads to an increased point radius of the cutting edge and results in a deterioration of the surface quality of the material processed, which frequently constitutes a criterion of the tool wear. To increase the durability of tools used in wood processing, it is justifiable to use wear resistant coatings, which need to exhibit high hardness and good adhesion while at the same time preserving sharp cutting edges of the blade. Based on a literature analysis conducted in studies [18-21], it was demonstrated that the properties of diamond-like coatings DLC may be applied for tools for the processing of wood or wood based materials, in order to increase their durability. The research results presented in the study [18] of amorphous carbon coatings, which are referred to in literature also as DLC [23], constitute a mixture of the ta-C phase and the graphite-like phase. During the tests described in the study [18], optimum values were determined of the selected parameters of the production of DLC coatings with the Modified Cathodic Vacuum Arc (MCVA) method, which could guarantee obtaining their most favourable properties from the perspective of the use for coating high speed steel plane knives for the processing of wood and wood based materials. In the initial phase of the research, the chemical composition, adhesion, hardness, elasticity modulus, surface roughness and the wear rates of coatings and counterspecimens were verified. After an analysis of the results obtained, the adhesion of the coatings was considered to be an important property from the perspective of the modification of the surfaces of tools for wood processing. Adhesion of DLC coatings was accepted as a criterion for the optimization of the manufacturing parameters. It is characterized by the value of the critical load L_{C2} in the scratch test. Another criterion was hardness and the coating wear rate determined in frictional contact with ceramics in tribological tests. The impact of the selected parameters of DLC coating production on the accepted criteria of optimization with the use of the experience planning module according to the Taguchi method, with the use of orthogonal tables in the Statistica software were analyzed [18]. Based on the statistical analyses of the results of the tests of the coatings it was found, that in order to ensure high adhesion of DLC coatings to high speed steel substrates, the following structure of the coating is needed

– Cr sublayer of 0.3 μm and external 1.8 μm DLC layer. The deposition parameters were: argon pressure 0.25 Pa and floating potential. To obtain high hardness and resistance to abrasive wear, high values are to be used of the substrate polarization voltage -80 V and low argon pressure of 0.01 Pa. Depending on the production parameters used, it was found that it is possible to obtain DLC coatings in a wide range of hardness from 20 to 60 GPa, and with good adhesion to high speed steel substrates. The properties of DLC coatings produced with optimized parameters [18] demonstrated that it is possible to use them to increase the durability of tools used for the processing of wood or wood based materials. It was conducted a verification of this assumption and described it in the studies [19-21].

Based on the research results presented in the study [18], the parameters were determined of the deposition of DLC coatings with substantially diversified properties, with a 0.3 μm thick Cr sublayer, which is presented in the study [19]. Coatings marked DLC(L) with the highest adhesion characterized in the scratch test (high values of the critical load L_{C2} of over 40N), DLC(H) coatings with the highest hardness (ca. 60 GPa) and a high hardness to elasticity module ratio ($H/E = 0.14$), as well as DLC(S) coatings that are conventionally produced, with intermediate properties. The research results presented in the study [19] serve to confirm obtaining of DLC coatings with complex structures and properties. Industrial tests were conducted on uncoated and coated by DLC plane knives from HS6-5-2 high speed steel. They had significantly diversified properties, while processing a Medium-Density Fibreboard (MDF) laminated on both sides, and which constitutes the basic material in furniture production. Knives with DLC(S) coatings demonstrated the lowest blade wear (Fig. 8), which may be connected with the high hardness of the coating of 38.5 GPa and the ratio of $H/E = 0.12$ and the average value of the critical load $L_{C2} = 38.5$ N and the highest average value of the critical load $L_{C3} = 45$ N determined during the scratch test, which proves their good adhesion to high speed steel knives. Near knife edges with DLC(S) coatings, after production tests, there occur the smallest crumbling and chips of the coating, which also serves to confirm their high adhesion. Knives with DLC(L) and DLC(H) type coatings demonstrated wear that was by ca. 20% high than uncoated tools (Fig. 8). A high wear of knife blades with DLC(H) type coatings, in spite of the lowest values of the wear rate determined in the tribological ball-on-disk test, may be the result of their significant brittleness and low adhesion characterized in the scratch test.

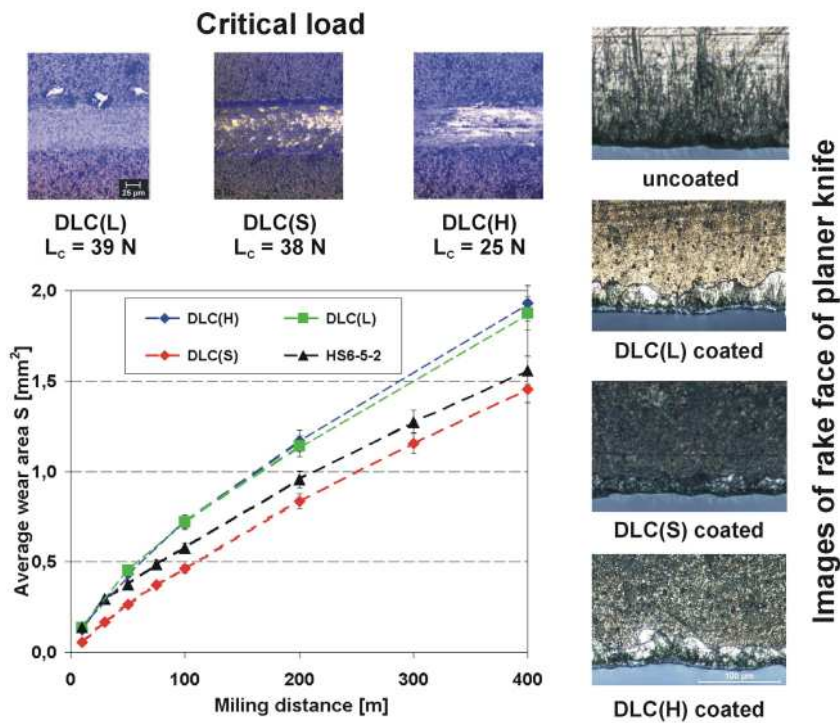


Fig. 8. Wear area S of the cutting edge in the function of the cutting length, wear images of rake face of cutting edge of planer knife and images of coating damage at critical load L_c in the scratch test. Based on [19]

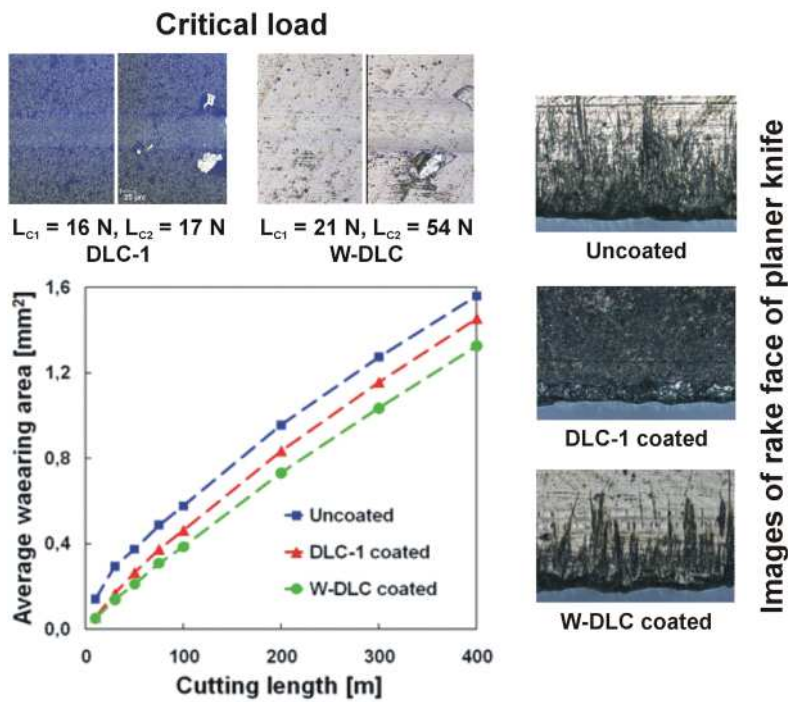


Fig. 9. Average wear area of the cutting edge in the function of the cutting length, wear images of rake face of cutting edge of planer knife and images of coating damage at critical load L_c in the scratch test. Based on [20]

This was also manifested by the losses of the pieces of the coating by the cutting edge of the knife and a significant rounding point of the blade. High hardness of 57.1 GPa and a high value of the ratio of $H/E = 0.14$ demonstrates a significant resistance of DLC(H) coatings to plastic strain, yet it does not indicate their ability to elastic strain with a simultaneous resistance to cracking. A high wear of knives with DLC(L) type coatings, even though they possessed high adhesion, determined in the scratch test ($L_{C2} = 40$ N), may be the result of their relatively low hardness of 22.7 GPa, and the related little resistance to abrasive wear, which may be confirmed by the abrasive nature of the wear of the edges of the blade with these coatings that is visible during microscope observations (Fig. 8) [19]. The nature of the work of plane knives, during MDF board processing in particular, results in a high mechanical load and thermal load of the blades. There occurs plastic strain of the tool substrate with a hard DLC coating deposited, which may cause it to crack, become damaged by chips, especially if the coating adhesion is low. This effect is caused by chips that flow down along the rake face which, by catching by the coating edges in the points of its discontinuity cause fragments to be torn away, all the more intensely when the coating adhesion is poorer that is characterized in the scratch test.

In the study [20], the results of the tests of plane knives with DLC coatings produced used in the processing of wood and wood based materials are described. TEST 1 was related to plane knives from high speed steel with DLC-1 and W-DLC coatings used in the processing of the MDF board with the cutting speed of 36 m/s. The coatings with the DLC-1 symbol consisted of a DLC layer with a Cr sublayer. They were produced using identical parameters as for the DLC(S) coatings previously described in the study [19], which demonstrated the highest durability during production tests. W-DLC coatings that were produced using the impulse reactive magnetron sputtering method, were composed mainly of a-C:H type amorphous matrix and tungsten carbide WC_{1-x} phase dispersed in it in the form of small precipitations sized 1-2 nm [20]. From the perspective of the application for coating tools for the processing of wood and wood based materials, W-DLC demonstrate favourable properties: hardness of 18.6 GPa, high resistance to cracking, and good adhesion (L_{C2} over 50 N), which was obtained by using between the coating and the steel substrate of a transition zone based on chromium and tungsten. Plane knives with DLC-1 and W-DLC coatings produced demonstrate smaller wear of the blades in relation to uncoated steel knives during MDF processing (Fig. 9). In spite of the hardness value of DLC-1 coatings (40.8 GPa) being over twice as high in comparison with W-DLC

coatings (18.6 GPa), their poorer adhesion, characterized in the scratch test ($L_{C2} = 17$ N), is one of the main reasons of a lower durability of coated knives used for MDF processing. The microscope observations of the rake faces of the knives after production tests indicate an abrasive nature of the wear of blades with W-DLC coatings (Fig. 9), similar to that of uncoated steel knives, which may be the result of a relatively low hardness of these coatings [20]. On knives with DLC-1 coatings produced, there are no visible traces of wear, which proves their high resistance to abrasive wear being the result of a high hardness of these coatings (40.8 GPa), while the visible fractures and chippings of the coating from the rake face of the knives, near the cutting edge, confirm their substantial brittleness and poor adhesion (Fig. 9). In order to improve the adhesion of DLC layers to high speed steel substrates, a sublayer based on chromium and chromium nitride Cr/CrN/Cr was developed; this type coating was marked as DLC-2 [20]. In the scratch test, which characterizes the adhesion of DLC-2 coatings to high speed steel, a critical load was obtained of $L_{C2} = 38$ N. Comparative tests were conducted for plane knives from high speed steel with DLC-2 and W-DLC coatings produced, in production conditions, for pine wood processing with the cutting speed 50 m/s (TEST 2); the test results are described in the study [20]. The wear of the plane knives, characterized by the blade wear area in relation to 1,000 running metres of the material processed, for knives with the W-DLC coating was lower by 10%, and for knives with the DLC-2 coating, it was lower by 25% as compared to the wear of uncoated knives from HS6-5-2 steel (Fig. 10). Microscope observations of the blades after production tests prove that the coatings protect the knives against abrasive wear (Fig. 10). Small chippings and peels of DLC-2 coatings, near the cutting edge only, demonstrate their good adhesion to steel substrates, which was obtained owing to the developed modification of the sublayer based on chromium and chromium nitride. DLC-2 and W-DLC coatings were produced also on shape knives made from sintered carbide, used in the processing of a glued floor board with the cutting speed of 78 m/s – TEST 3 [20]. The results of the industrial tests of tools, which were conducted to the point of their critical wear, demonstrate that the durability of tools with W-DLC and DLC-2 coatings is substantially higher (by 200 and 300% respectively) in relation to uncoated tools from sintered carbides (Fig. 11). Carbon coatings that are used on tools, especially multilayer DLC-2 coatings, reduce their abrasive wear, and they also improve the work of tools by reducing adhesion of glues and resins to the blades that are contained in the non-homogenous workpiece; simultaneously they improve the quality of the workpiece.

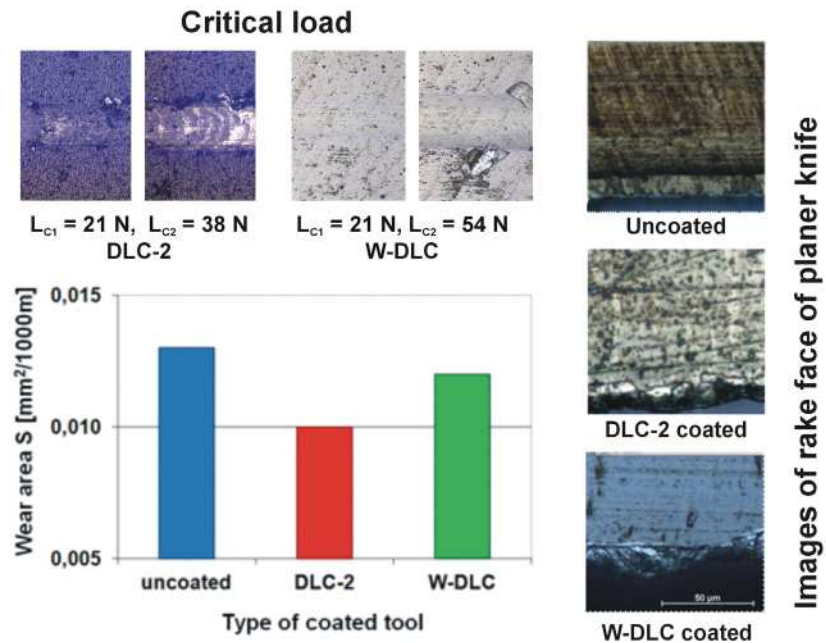


Fig. 10. Average cutting edge wearing area S on milling distance 1000 m, wear images of rake face of cutting edge of planer knife and images of coating damage at critical load L_C in the scratch test. Based on [20]

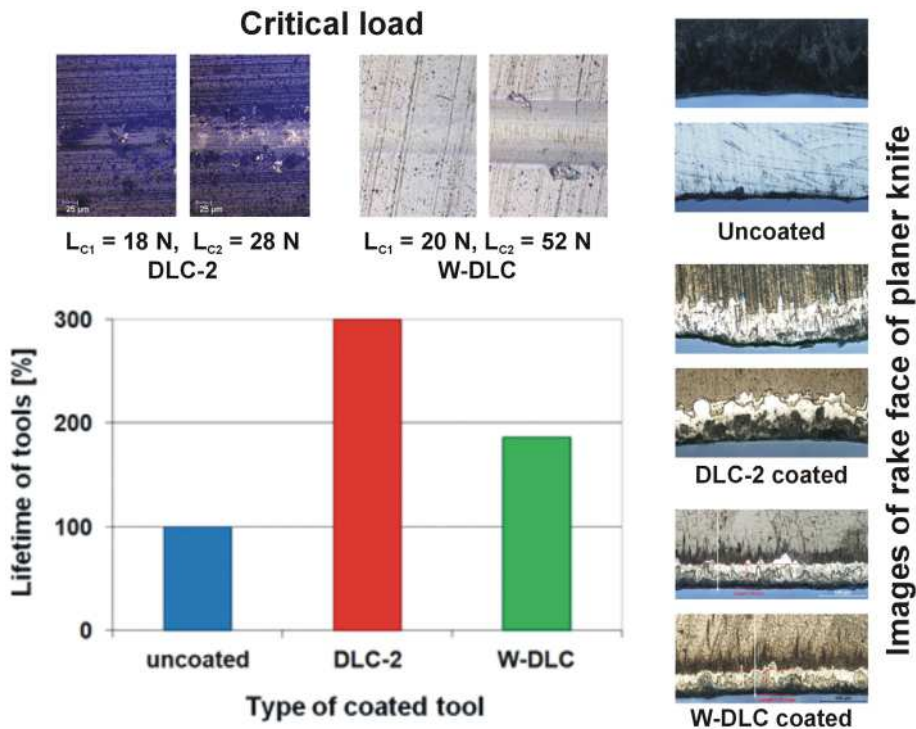


Fig. 11. Lifetime of DLC-2 and W-DLC coated and uncoated WC-Co blades, images of rake face of knife (groove forming – upper image, tongue forming – bottom image) after operational wear testing and images of coating damage at critical load L_C in the scratch test. Based on [20]

Summary related to the production technology of coatings with the top DLC coating, the results of tests related to the properties of these coatings as well as the results obtained of industrial tests of coated tools. Owing to the use of the Taguchi optimization method, a lot of important information was obtained related to the impact of the deposition parameters on the properties of DLC coatings with a relatively small number of experiments [18]. The thickness of the DLC coating and of the Cr sublayer is of a significance to the adhesion of coatings. In order to ensure high adhesion of coatings to substrates from HS6-5-2 high-speed steel, a thick Cr sublayer (0.3 μm) and DLC coating (1.8 μm) produced with high argon pressure (0.25 Pa) need to be used; no polarization of substrates is to be used: floating potential. The value of the polarization tension of substrates and argon pressure are of a greatest significance to the hardness and wear by friction for DLC coatings. In order to obtain high hardness H and resistance to abrasive wear, high values of the substrate bias voltage (-80 V) and low argon pressure (0.01 Pa) are to be used. Owing to changes in argon pressure and the substrate bias voltage during the deposition of coatings with MCVA technology, it was possible to obtain DLC coatings with the content of bindings sp^3 ranging from 30% to 60% and significantly differing properties [8,9]. Hardness changed in the range from 22.7 to 57.1 GPa, and the proportion of H/E from 0.09 to 0.14. In tribological tests, all the coatings were characterized by low wear. The adhesion of the coatings determined by critical load L_{C2} in the scratch test ranged from 22 to 40 N. Adequately selected conditions of the deposition and obtained properties of the coatings may have an influence on an extension of durability of coated plane knives in relation to uncoated steel tools, when using them to process a wood based material, that is a laminated MDF board [19,20]. The reason of the highest durability of tools coated with DLC(S) may be intermediate values of the relation of hardness to the modulus of elasticity and the best adhesion determined in the scratch test by the highest values of the critical load L_{C3} of over 45 N. In the processing of wood-based materials (MDF, floor board), it is usually tools made from sintered carbides or from tool ceramics, largely not coated, that are used. Considering the brittleness of tools from sintered carbides and tool ceramics, it was demonstrated that the use of tools from high speed steels with hard coatings of DLC and W-DLC, may constitute an alternative in wood-based materials processing. A technology was developed of the production of coatings with the top DLC layer of a high hardness with sublayers based on chromium that ensure to them high adhesion

to tools from high speed steels and sintered carbides for the processing of wood and wood-based materials. The results of the industrial tests of tools coated with these coatings, demonstrate an increase of their durability, in particular during processing with high cutting speeds of non-homogenous wood based materials that contain organic binding and hardening compounds [19,20].

A noticeable increase of the durability of tools with DLC coatings produced during the processing of wood and wood based materials [19,20] and problems with the adhesion of these coatings constituted the reason for a study and tests of coatings with the top DLC coating with three types of sublayers based on chromium, which is described in the study [21]. The influence of the modification of the rake face of plane knives from HS6-5-2 high speed steel with this type coatings produced, on their durability and wear during industrial tests in the processing of pine wood was determined (Fig. 12). In relation to uncoated knives, the knives with the Cr/DLC coating demonstrate about 8% lower wear. For knives with the Cr/CrN/Cr/DLC multilayer coating the wear is ca. 23%, and for knives with the modular Cr/(CrN/CrC)/Cr/DLC multilayer coating is by ca. 2.5 times smaller. On the rake face of uncoated knives, the traces of wear at the distance up to ca. 100 μm from the edge are visible. Rounding of the knife edge is also observed. For knives with the Cr/DLC coating, crumbles of the edge with losses of the coating are visible at a distance up to 50 μm from the cutting edge. Knives with the Cr/CrN/Cr/DLC coating possess crumbles of the edge with losses of the coating at a distance up to 30 μm from the edge.

Knives with the Cr/(CrN/CrCN)/Cr/DLC coating show the smallest wear; there is some marginal crumbling of the edge and small chips of the coating at a distance up to 20 μm from the edge (Fig. 12). Lack of visible traces of wear on the rake face of all the coated knives demonstrates the fact that coatings with a hard external DLC layer (ca. 34 GPa) and chromium based sublayers, especially of Cr/(CrN/CrCN)/Cr type, perform a favourable anti-wear function. Multiple Cr/(CrN/CrCN)/Cr/DLC coatings produced on plane knives from HS6-5-2 steel demonstrate the lowest adhesion $L_C = 27$ N, which is characterized in the scratch test in comparison to Cr/DLC ($L_C = 39$ N) and Cr/CrN/Cr/DLC ($L_C = 37$ N) coatings. However, Cr/(CrN/CrCN)/Cr/DLC coatings demonstrate the smallest wear during planing of pine wood (Fig. 12) [21]. This may be the result of multi-layer and modular structure of these coatings [24], which improves the resistance to dynamic stresses, which prevents cracking propagation that occur when the coating is damaged near the cutting edge

during cutting. These coatings have an influence on a reduction of the wear of the coated knives and a substantial extension of their durability, which permits work with no

stoppages for the replacement of tools. This leads to a reduction of costs related to unforeseen stoppages of the production lines in continuous production.

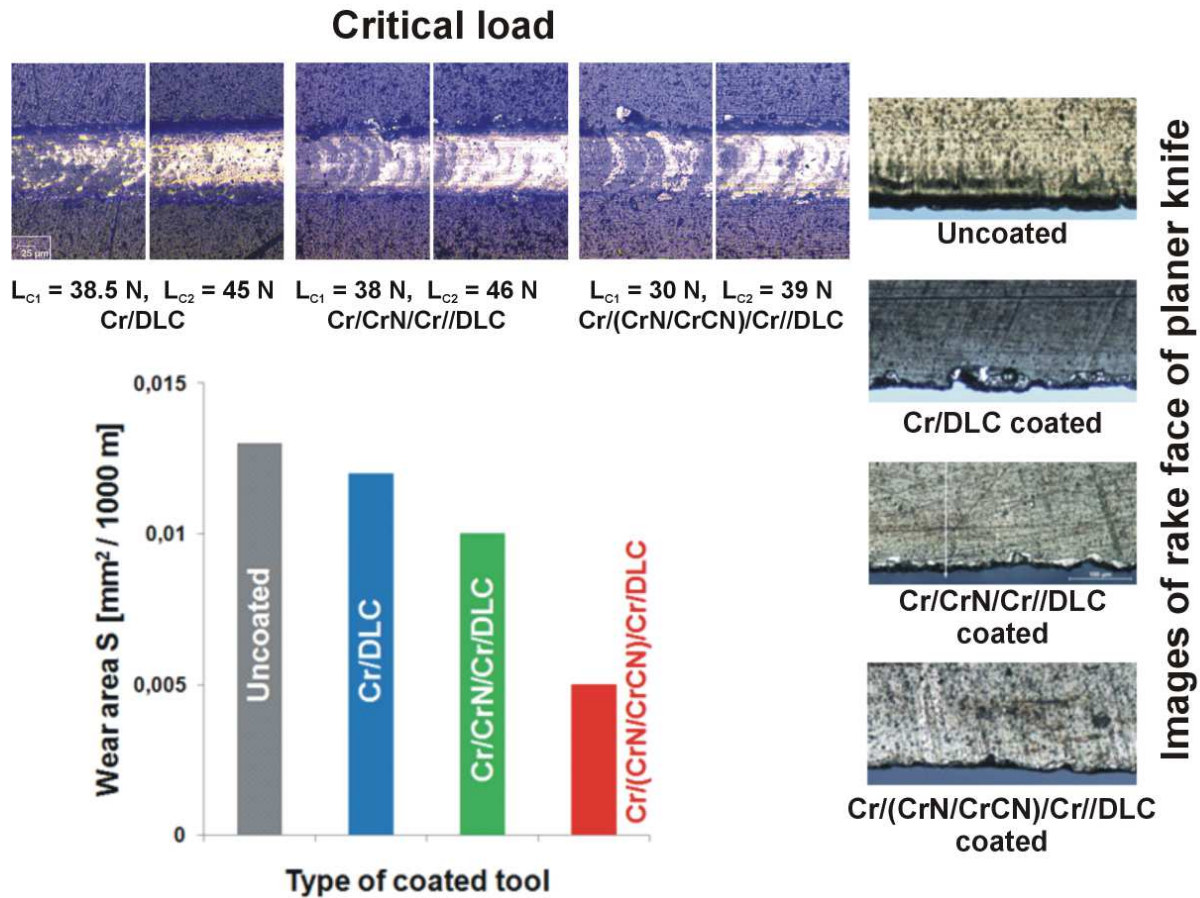


Fig. 12. Average cutting edge wear area S on milling distance 1000 m, wear images of rake face of cutting edge of planer knife and images of coating damage at critical load L_C in the scratch test. Based on [21]

4. Conclusions

The characteristics of the adhesion of coatings through the determination of the value of the critical load in the scratch method provides information on the level of practical adhesion. During the determination of the critical load in the scratch test that characterizes the adhesion of the coatings, there occur substantial unit pressures that are similar to those on the blade during cutting. The results of this test reflect not only the adhesion durability in the coating-substrate area but also the impact of all the stresses that occur there.

Based on the results of the examinations of adhesion through the scratch test, it is possible to quickly obtain

an assessment of the usefulness of the coatings produced on cutting tools. Nevertheless, this cannot be the only and final method of such evaluation. A direct comparison of the results of the scratch tests of coatings is possible when adhesion is being examined of different coatings yet on the same type of the substrate. The results may be compared when we are examining adhesion of identical coatings produced on similar substrates that undergo different process of the preparation of the surface. Also, coatings may be compared that are deposited with the aid of different technologies, yet only when the substrates are from the same material and the coatings possess the same chemical composition and similar structures.

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