Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 33
ISSUE 1
March
2009

Investigation of the life-time of drills covered with the anti-wear Cr(C,N) complex coatings, deposited by means of Arc-PVD technique

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Received 12.02.2007; published in revised form 01.03.2009

Manufacturing and processing

ABSTRACT

Purpose: This research was done to study the tribological behavior of twist HS6-5-2 drills covered with Cr/CrN/CrN+Cr_2N/Cr(C, N) and Cr/CrN/ Cr(C, N) coatings as well as to obtain detailed information about microstructure and properties of investigated coatings.
Design/methodology/approach: The coatings were deposited by the ARC-PVD process and examined revealing their microstructure: with the use of SEM and TEM microscopy and composition: chemical (EDS microanalysis) and phase (XRD). Also microhardness, Young's modulus and adhesion were measured using NHT CSEM hardness tester and scratch tester.
Findings: The electron energy disspersive (EDS) analysis of coatings showed higher concentration of nitrogen (in the internal coating-zone) and carbon (in the external coating-zone). The X-ray diffractometry showed that coatings are mainly composed of CrN and Cr_2N nitrides. The TEM analysis revealed that coating crystallization starts with nanometric Cr_2N nitrides coherent with the ferrite matrix grains. The coatings turned out to improve the lifetime of the drills (much better than that of un-coated ones).
Practical implications: The main aim of this work is to determine properties of multilayer and multi-component coatings on basis of Cr, C and N, which are potential candidates for replacing TiN coatings in certain applications.
Originality/value: The paper contributes to better understanding of microstructure and properties of the coatings (chemical and phase composition, microhardness, Young's modulus, adhesion), as well as lifetime of the coated drills.
Keywords: Cr(N,C) coatings; Arc-PVD; Drilling test; Scratch-test; Wear; Microstructure; EDS

Reference to this paper should be given in the following way:
J. Kusinski, M. Rozmus, J. Bujak, Investigation of the life-time of drills covered with the anti-wear Cr(C,N) complex coatings, deposited by means of Arc-PVD technique, Journal of Achievements in Materials and Manufacturing Engineering 33/1 (2009) 86-93.

1. Introduction

The use of thin, physically hard vapour deposited (PVD) coatings in tribological applications has become more and more widespread [7, 9, 13]. Tools and machine element working surfaces, covered with such thin, wear resistant coatings usually exhibit improved service behaviour and increased lifetime [2-5, 8, 10-12]. One of the PVD techniques, which develops dynamically due to its particular possibilities is cathodic arc PVD process [1, 2].

The effectiveness of surface protection depends on proper selection of the coating chemical composition, its macro- and microstructure, as well as coating mechanical properties in relation with coated material and working conditions of elements being in contact. The most frequently used materials for wear resistant coatings in tribological applications are nitrides, carbides nitro-carbides, borides and oxides of rare-earth elements.

Explicit selection of the coating material for protection of cutting tools made of high-speed tool steel used to cut steels and alloys are a complex problem. It is because the exploitation properties of coatings are determined not only by its properties but also by properties of the coating-substrate system [10, 12].

Ideal tribological coating for cutting tools should be characterized by neutral reactivity of external zone with cut material, high hardness of the central zone and good adhesion of the internal zone to the substrate. It is practically impossible to achieve such properties in the case of monolithic coatings. For example, increasing hardness and strength usually reduces coating ductility and its adhesion to the substrate [12]. Many more possibilities can be achieved in case of using multilayer coatings. Use of a multilayer coating structure gives a possibility to differentiate properties on the coating crossection by changing its chemical composition and structure of subsequent layers, as well as quantity, thickness and sequences of deposited layers. A characteristic structural feature of such multilayer coatings is presence of interlayer transition zones – interlayer boundaries. Presence of such interfaces between the individual layer results in an increase in hardness and strength of the coating. It may also change the mechanism of coating destruction from one stage to multistage. In case of the monolayer coatings their destruction initiates usually at the external and/or at the coating/substrate surfaces and cracks easily pass through the coating [10].

The Arc-PVD technique is relatively simple method and reliable technology which permits to deposit different kinds of coatings [16]. The most significant advantage of the Arc-PVD process is ability to deposit, at relatively low temperatures (even below 200^{o}C), coatings of good adhesion to the substrate. This is due to the uniform and highly energetic ionic bombardment of substrate. Indeed, the substrate surface is changed microscopically to a depth of a few nanometers what creates ideal growth conditions for coatings. Uniform ion bombardment during coating leads to a favorable stress pattern in the hard coatings so that flaking and micro-cracks can be avoided [15]. Since, the Arc-PVD process permits to get high quality coatings at low bias voltage and at temperatures (<200^{o}C), many grades of steels (cold worked steels) can be coated using that process without decreasing their hardness produced during earlier heat treatments.
In case of multilayer coatings, crack development stops at the interlayer boundaries. The example of such complex coatings can be: Cr/CrN/Cr(C,N) and Cr/CrN/CrN+Cr_2N/Cr(C,N). It is well known that coatings made on the basis of Cr, N and C characterize high hardness, wear resistance, good corrosive and oxidation resistance at high temperatures and it's the reason why multilayer CrN coatings are potential candidates for replacing of TiN or Ti(C,N) coatings in certain applications.

The aim of this work is to present the results of an investigation of behavior of HS6-5-2 high-speed steel twist drills covered with different kinds of Arc-PVD multi-layer coatings (basing on Cr, N and C elements) during drilling of steel plates.

2. Experimental details

2.1. Sample preparation

The investigated Cr/CrN/Cr(C,N) – type A, Cr/CrN/CrN +Cr_2N/Cr(C,N) – type B multilayer and multicomponent Cr-C-N – type C coatings were deposited in the Institute for Terotechnology in Radom, using the Arc- PVD technique (Bujak *et al.* 2004). The industrial multi-source coating device, type NNW 6 614, was used for the deposition process. The coatings were deposited on commercial HS6-5-2 high speed steel (ϕ = 6 mm) twist drills (Fig. 1) and on mirror polished (R_a=0.01 mm) 27 mm in diameter and 10 mm thick disks (Fig. 2) made of hardened and tempered HS6-5-2 steel (64 HRC) and soft Armco iron. All substrates (drills and discs) were ultrasonically cleaned in the alkaline solution and rinsed in distilled water. Prior to the coating process, the substrates were preheated to the temperature of 200 °C by a resistance heater and then cleaned by the arc enhanced glow discharge etching process. In the second etching step, the substrates were bombarded with arc-evaporated Cr ions at the negative bias voltage of -1000 V. During deposition the substrates were negatively biased and kept at the temperature range of 150-250^{o}C throughout the process. The coats were grown to a thickness of approximately 4 μm. Desired structural construction of the coatings have been obtained by using variable gas mixture (C_2H_2, N_2, Ar), gas pressure in the working chamber ($1x10^{-4}$-$3.5x10^{-2}$ [mbar]), sources current - 75 A, substrate bias voltage (-150 - -250 V) and substrate temperature (320 - 370 ^{o}C). All parameters of the coatings deposition are present in Table 1. As it is shown in drilling, the tool life increases linearly with thickness of the coating [14]. In case of our experiments, optimal value was chosen to be about 4-5 μm.

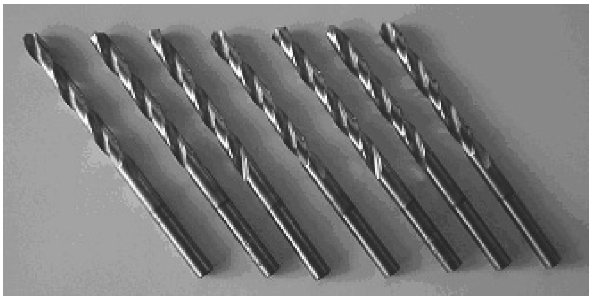

Fig. 1. Image of the twist drills coated with Cr/CrN/CrN+Cr_2N/Cr(C,N) coating

Table 1.
Parameters of the coatings deposition

Type	Composition of gax mixture [ml/min]	Gas pressure in the working chamber [mbar]	Sources current [A]	Substrate bias voltage [V]	Substrate temperature [^{O}C]
A	-	1 x 10^{-4}	75	-150	340
	N_2 – 490	3.5 x 10^{-2}	75	-250	390-330
	C_2H_2 - 62 N_2 – 188 Ar - 178	7 x 10^{-3}	75	-150	330-360
B	-	1 x 10^{-4}	75	- 150	340
	N_2 – 475	3.5 x 10^{-2}	75	- 250	390-335
	N_2 – 280	2.5 x 10^{-3}	75	- 250	340-370
	C_2H_2 - 62 N_2 – 188 Ar - 175	7 x 10^{-3}	75	- 150	360-350
C	C_2H_2 - 62 N_2 – 188 Ar – 170	7 x 10^{-3}	70	- 150	320-330

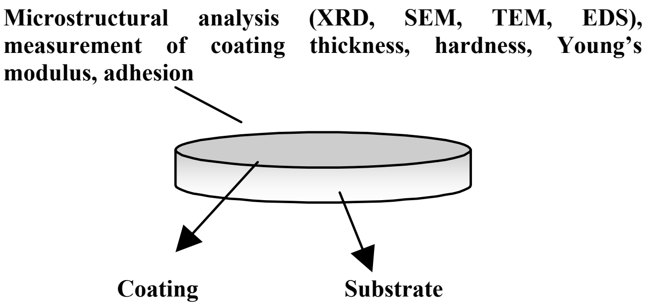

Fig. 2. Schematic drawing show the witness (HS6-5-2 steel or Armco iron) sample coated with Cr/CrN/Cr(C,N), Cr/CrN/CrN+Cr_2N/Cr(C,N) or Cr-C-N and variety of tests performed for complete characterization of the coating

2.2. Coatings characterization

The complete characterization of the investigated Cr/CrN/Cr(C,N), Cr/CrN/CrN+Cr_2N/Cr(C,N) and Cr-C-N coatings was performed on the witness HS6-5-2 steel and Armco iron samples that were coated together with drills (Fig. 2). The structural construction of the investigated coatings is presented in Fig. 3. The coatings thickness was determined by the calotte test method. The hardness and Young's modulus were investigated using the nanoindentation method (Nano-Hardness Tester NHT CSEM equipped with a Berkovich indenter loaded with velocity: V = 40 nN/min to reach maximal penetration: h_{max} = 200 nm) Hitachi-3500N scanning electron microscope (SEM) was used to examine the coating morphology and to monitor the changes of the drill cutting edges during the drilling test and to identify the type of wear mechanisms.

The coatings structure was determined by applying X-ray BRUKER D-8 diffractometry with Co-K radiation and planar transmission electron microscopy (TEM) investigations of thin foils. An analysis of the elemental composition of the coating on spherical marks made with a Gatan dimple grinder through the coating was performed using Noran energy depressive X-ray spectrometry (EDS). The adhesion of the investigated coatings with the substrate was evaluated by scratch testing with the CSEM Revetest instrument.

Standard scratch parameters of d *L*/d *t*=100 N/min and *v*=10 mm/min were used.

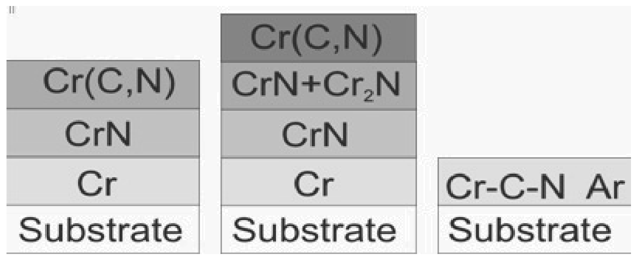

Fig. 3. Schematic diagrams showing structural construction of the tested: Cr/CrN/Cr(C,N)-type A, Cr/CrN/CrN+Cr_2N/Cr(C,N) – type B and Cr-C-N – type C, coatings

2.3. Drilling tests

Dry drilling tests were carried out using a vertical milling machine WKA-25 working at constant rotation speed of 500 rev/min and a feed speed of 0.6 mm/rev. A plate with dimension of 300 x 300 x 30 mm (annealed steel of composition: 0.18%C, 1.4%Mn, 0.3%Si, 0.2%Cr with hardness of 159 HV) was used as a work piece material. For the reference to the drills covered with Cr/CrN/Cr(C,N) and Cr/CrN/CrN+Cr_2N/Cr(C,N) coatings, the un-coated drills were also tested. A continuous sequence of holes were machined until the drill reached the end of its life. The catastrophic failure (breakage, loss of cutting capacity or cutting edge deformation) was applied as a drill life criterion. After a defined number of drilled holes (1, 5, 10, 20, etc) the worn drills were analyzed with a scanning electron microscope to monitor the changes of the drill cutting edge and to identify the type of wear mechanisms. The areas of detailed SEM analysis of the drill cutting edge are marked in Fig. 4. The drill life was calculated as the mean value of three tests.

3. Results and discussion

3.1. Characterization of coating parameters

Table 1 presents, results of measurements of the coating thickness, hardness, Young's modulus and number of holes drilled to the moment of catastrophic failure. Measured values of the coating thickness were 4.3 and 4.4 µm, respectively for coating types A and B. The thickness of the component layers of Cr/CrN/Cr_2N+CrN/Cr(C,N) coating was: Cr – 0.1 µm, CrN/Cr_2N – 2.1 µm and CrN/Cr(C,N) – 2.2 µm, Cr-C-N – 2.6 µm.

Hardness and Young's modulus measurements show the highest values for multi-component Cr,C,N coating: 2321 HV, 356 GPa and higher for Cr/CrN/Cr(C,N): 2179 HV, 338 GPa than for Cr/CrN/Cr_2N+CrN/Cr(C,N): 2112 HV, 311 GPa, coating. The number of holes drilled to the moment of catastrophic failure was higher for Cr/CrN/Cr(C,N) than for Cr/CrN/Cr_2N+CrN/Cr(C,N) coated drills. Drilling ability of both coated drills was much better than that of uncoated ones. Analysis of chemical composition of the coatings have been performed by using of EDS Noran microprobe on Armco iron and HS6-5-2 steel witness sample. Fig. 5a shows an SEM image of the coating crossection made using the dimple grinder and EDS spectrograms recorded in points 1, 2 and 3 (Fig. 5b).

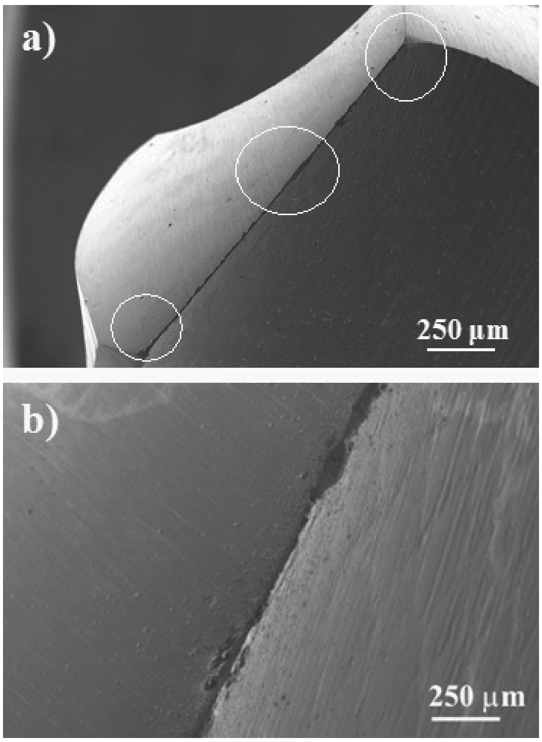

Fig. 4. SEM image of the drill, with marked areas of SEM examinations (a), cutting edge of the drill (b)

Table 2.
Results of the hardness, Young's modulus, coating thickness, as well as measurements of hole numbers drilled to the catastrophic failure.

Type of coating	Hardness [HV]	Young's modulus [GPa]	Coating thickness [µm]	Number of drilled holes
A	2179±57	338±7	4.3	230
B	2112±132	311±13	4.4	220
C	2321±75	356±42	2.6	-
Uncoated drills	820	-	-	150

Linear EDS analysis of the Cr/CrN/Cr(C,N) coating deposited on HS6-5-2 steel presented in Fig. 6 confirmed that the nitrogen concentration in near substrate layers is higher than the concentration of carbon. It is in accordance with the provided coatings structure.

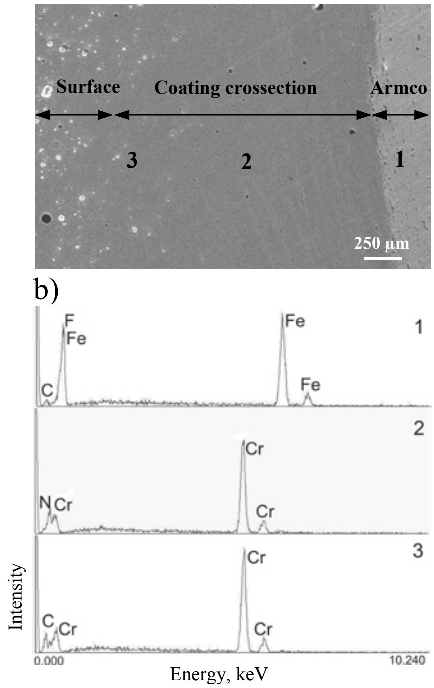

Fig. 5. SEM microphotograph showing crossection the Cr/CrN/CrN+Cr_2N/Cr(C,N) with marked areas of EDS microanalysis (a) and EDS spectra recorded in points 1, 2 and 3 (b)

XRD analysis was conducted with the use of classical Bragg-Brentano geometry, as well as in SKP geometry using variable X-ray incident angles 1, 3, 6 and 9 degree. Such tests allowed analysis of the coating structure at different levels. At low degrees only Cr_2N and cubic CrN peaks are present from reflections representing the substrate phase peaks, which were present when classical XRD analysis was performed (Fig. 7a, b,c).

SEM analysis of the broken samples showed that delamination of the coating from the substrate and between sub-layers may occur (Fig. 8).This observation is in accordance with the observation made during the scratch test (Fig. 9). We observed that with an increased load during the scratch test (about 14N), destruction of both type of coats starts due to elastic and plastic deformation of the coating. Destruction is initiated by cracks and delamination of the external Cr(C,N) layer, with larger loads leading to cracking in deeper layers and their delamination. However, when loads reaching 200 N the coating is completely removed from the surface. The advantage of the multilayer coating as compared to the monolayer is that crack development in one particular sub-layer stops at the interlayer boundaries, which may increase coating life-time (PalDey and Deevi, 2003).

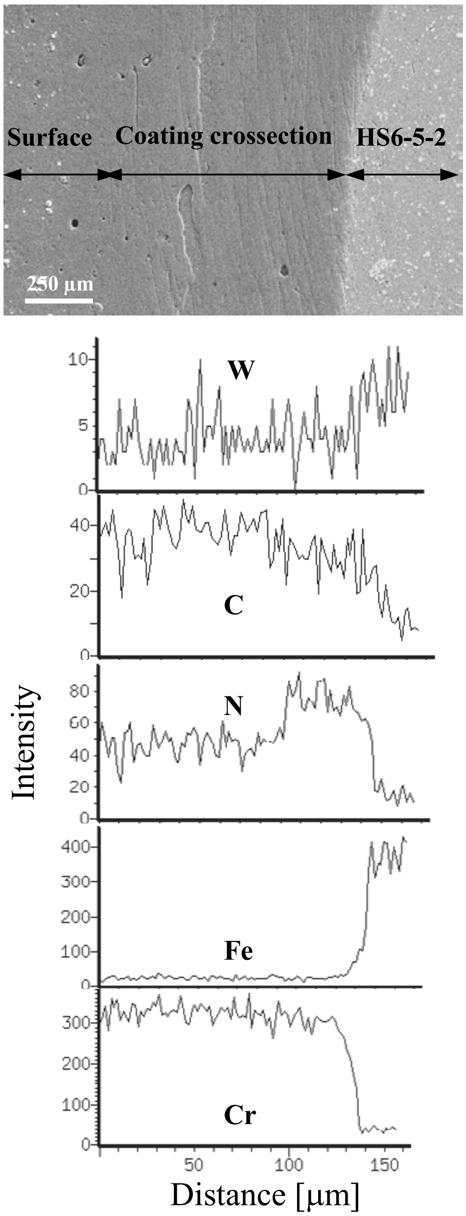

Fig. 6. The SEM microphotograph showing crossection the Cr/CrN/Cr(C,N) coating and results of EDS linear analysis of W, C, N, Fe, Cr

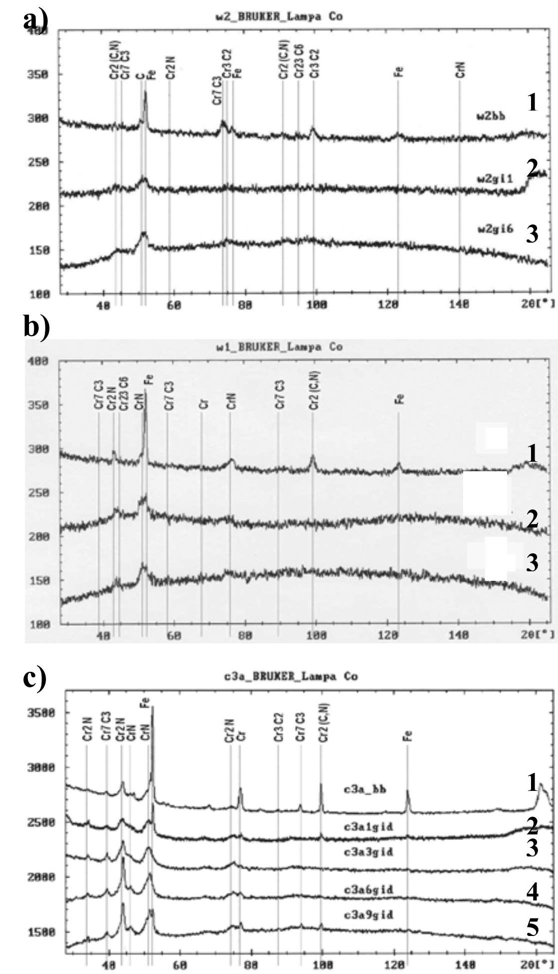

Fig. 7. XRD spectrograms Cr/CrN/Cr(C,N), Cr/CrN/CrN $+Cr_2N$/Cr(C,N) and Cr-C-N coatings obtained by using of classical Bragg-Brentano geometry (1) and SKP geometry at $\alpha=1^0$, 3^0 , 6^0, 9^0 (adequately 2, 3, 4 and 5)

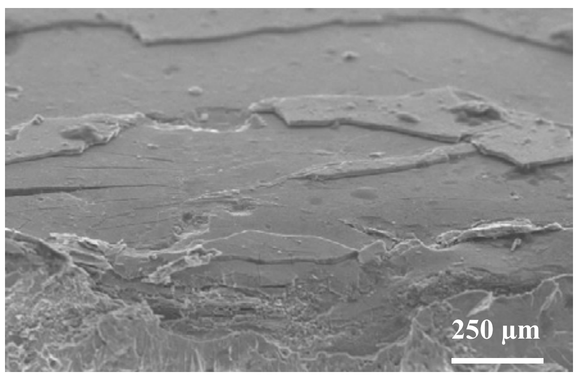

Fig. 8. SEM image of the fracture of Armco with Cr/CrN/CrN+Cr_2N/Cr(C,N)

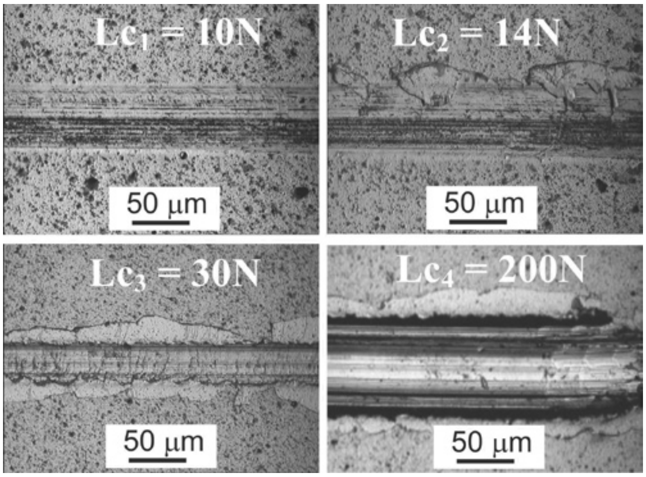

Fig. 9. Micrographs showing the scratch-paths at the surface of Cr/CrN/CrN+Cr_2N/Cr(C,N) coating

3.2. Results of investigation the coatings surface

The SEM investigation of surface topography of coatings indicate the presence of numerous defects in structure, such us: droplets and defects of forming during the crystallites growth (Fig 10).

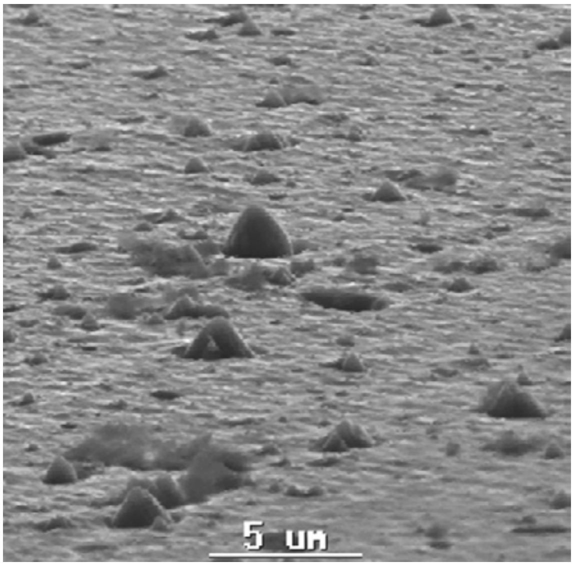

Fig. 10. SEM micrograph showing the surface topography of Cr/CrN/CrN +Cr_2N/Cr(C,N) coating

Micrographs (Figs. 11, 12) of the multilayer and multi-component coatings surface morphology showed that participation of defects on the surface of coatings depends on the coatings type and is higher for multilayer coatings. Higher percent od defects in multilayer coatings is causes by cumulation of defects generated during deposition of subsequent layers of coatings.

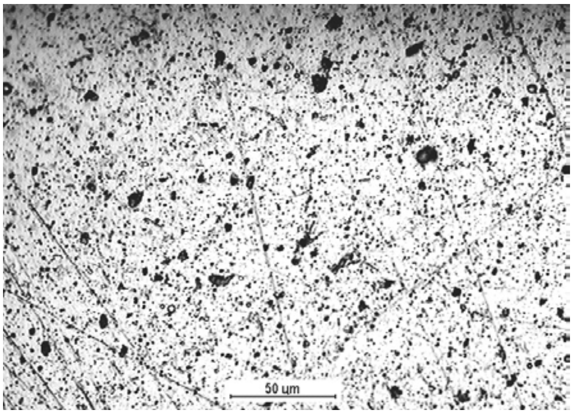

Fig. 11. Light microscopy image showing the surface morphology of Cr-C-N coating (magnification 500 x)

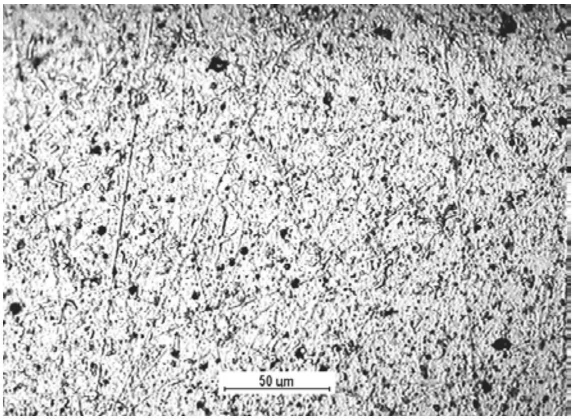

Fig. 12. Light microscopy image showing the surface morphology of Cr/CrN/CrN +Cr_2N/Cr(C,N) coating (magnification 500 x)

3.3. Microstructural characterization

Thin foils for TEM examinations were prepared from coatings that were deposited on an Armco iron substrate. Figs. 13a, b, c show planar TEM images of the microstructure of B coating.

TEM thin foils were prepared from different areas of the coating (a – near the substrate, b – central area and c- near surface area). Nitrides growing on three differently oriented ferrite grains <112>, <001> and <011> of the substrate were chosen for crystallographic analysis.

It seems that the coating growth starts by crystallization of ultra fine of 20-30 nm chromium nitrides (initially CrN, than Cr_2N and finally Cr(C,N). Due to oalescence of growing crystals, the nitride sizes (cross-sections) in the central and near surface areas are larger (however, smaller than 100 nm). The coherency

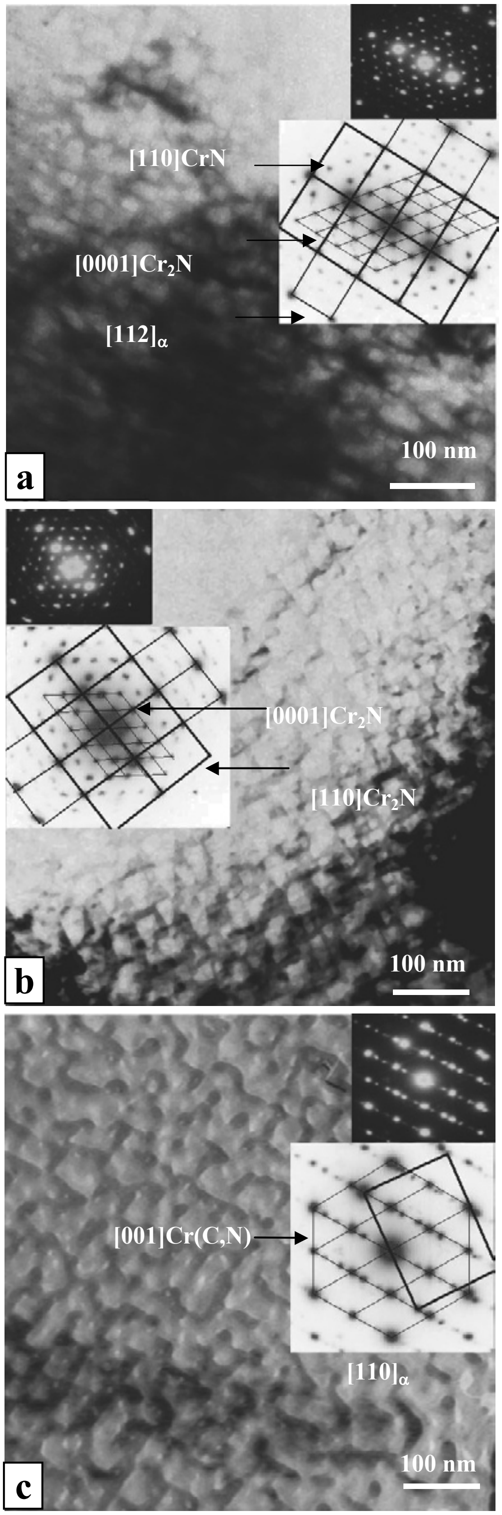

Fig. 13. The TEM analysis reviled that coating crystallization starts with nanometric Cr_2N nitrides coherent with the ferrite matrix grains; (a – near the substrate, b – central area and c- near surface area)

relationship between Cr_2N and alpha iron which is suggested by this examination can be expressed as: $[0110]_{Cr2N}$ || $[112]_\alpha$; $[1210]_{Cr2N}$ || $[002]_\alpha$ and $[1010]_{Cr2N}$ || $[110]_\alpha$. The characteristic feature of growing chromium nitrides is their regular rectangular or square like crossection. It was revealed that nitride crystals faces are parallel with <110> and <200> directions of the substrate alpha iron.

3.4. SEM analysis of the worn drills

Figures 14 and 15 are the SEM images of the drills (uncoated and coated with the coating of type A, respectively) both after catastrophic failure. In the experiment condition, coated drills with both coatings (type A and B) show better life for drilling that uncoated drills. Drilling tests and SEM observation showed that the degradation mechanism of the examined coated drills changes from adhesive at relatively low drilling velocity to diffusive and finally to intensive cracking of the substrate material. The sticking process of the worn material was intensive in case of uncoated drills (Fig. 14).

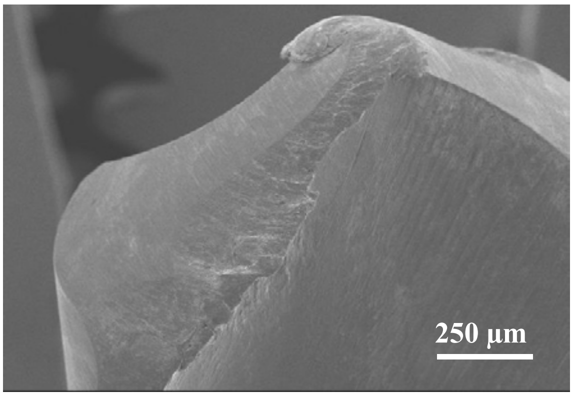

Fig. 14. SEM image of the uncovered drill after catastrophic failure (after drilling 120 holes)

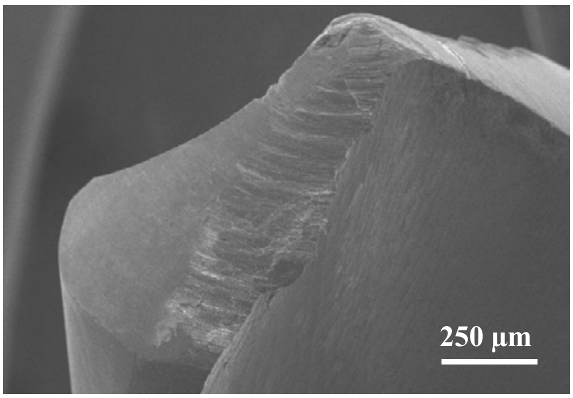

Fig. 15. SEM image of the covered drill after catastrophic failure (after drilling 230 holes)

4. Conclusions

The results obtained in this work suggest the following conclusions:

- The cathodic Arc PVD process is suitable to deposit a multilayer Cr/CrN/Cr(C,N), Cr/CrN/CrN+Cr_2N/Cr(C,N) and multicomponent Cr-C-N coatings
- The obtained results showed that both multicomponent Cr-C-N as well as multilayer Cr/CrN/Cr(C,N) and Cr/CrN/CrN +Cr_2N/Cr(C,N) investigated coatings possess high values of hardness and Young's modulus (within the range 2100 – 2350 HV, and 310 – 360 GPa) .
- Higher defects value in multilayer coatings is causes by cumulating process of defects generated during deposition of subsequent layers of coating.
- The life time of the Cr/CrN/Cr(C,N) coated drills is longer than that of drills coated with Cr/CrN/CrN+Cr_2N/Cr(C,N) and much longer than uncoated drills.
- XRD analysis confirmed the presence of expected phases: chromium, chromium nitrides and chromium nitro-carbides in both types of examined coatings.
- Using planar TEM examination of coatings, it was revealed that nitrides start to grow as a ultrafine crystals possessing coherency relationship with the substrate. During further coating growth, nitride coarsening (up to about 100 nm) occurs due to crystal coalescence.
- Both examined coatings present multistage mechanism of degradation typical for the multilayer coatings.
- Drilling test degradation of coated tools is mainly due to sliding and abrasive wear while in the case of uncoated drills adhesive sticking of worn materials may also occur.

Acknowledgements

The authors wish to thank Institute for Terotechnology and AGH-UST for technical support and Committee of Scientific Research of Poland for financial assistance (project No.: 11.11.110.566).

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