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The effect of surface pre-treatment and coating post-treatment to the properties of TiN coatings

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Abstract. Nowadays physical vapour deposited (PVD) coatings are widely used in a variety of different applications to reduce the wear of tools. Thin hard coatings have good wear resistance only when they have good adhesion and certain surface roughness. Thus, dry micro-abrasive blasting method with SiC abrasive was implemented as a pre-treatment process on WC-Co substrates. Post-treatment was conducted by using drag grinding method. The effect of surface pre- and post-treatment on the adhesion and tribological properties of TiN hard coatings have been investigated. TiN hard coatings were deposited by means of lateral rotating cathode arc method with the thickness of 2.3 µm. The surface roughness was measured by means of Mahr Perthometer and the standard Rockwell adhesion test was implemented to evaluate the adhesion of PVD TiN coatings. Tribological properties were determined by ball-on-disk method. The experimental part was focused on the effect of microblasting on the tribological properties of the coating. The relation between post-treatment of the coating and friction coefficient is indicated.

Key words: PVD coatings, surface roughness, surface pre-treatment, coating post-treatment, adhesion.

1. INTRODUCTION

The wear of tools has great economical effect on production due to tool and maintenance costs. Hard thin physical vapour deposited coatings are used to reduce abrasive, adhesive, erosive, sliding and fretting wear [1–4]. Wear resistance of the tool is related to the coating roughness (oiled condition) and adhesion of the coating [5–7]. Good adhesion of the coating assures better tool lifetime. In dry wear applications it is important to possess lower roughness and coefficient of friction (CoF) to guarantee wear resistance of tools [8,9]. Therefore, understanding the relationship between mechanical/tribological properties of coatings and methods of deposition and substrate preparation is of primary importance.

Usually tools are produced using milling, turning, electrical discharge machining (EDM) and grinding. The machining method could turn the tool surface unsuitable for coating and lead to unsatisfactory adhesion or surface roughness with insufficient lubrication properties [10,11]. Using surface pre-treatment and coating post-treatment it is possible to influence the properties of the substrate–coating system like optimization of stresses in the ground surface, roughness, adhesion and tribological properties [12,13].

The aim of this study was to analyse the effect of WC-Co substrate surface pre-treatment and PVD coating post-treatment on surface roughness, adhesion and tribological properties as CoF, etc.

2. EXPERIMENTAL

Grinded WC-10Co hardmetal substrates with the size of 25 × 15 × 3 mm, with surface roughness $R_a = 0.05$, 0.2, 0.4 and 0.8 were used. Substrate samples with $R_a = 0.05$ were produced by the dry micro-abrasive blasting (MAB) treatment. The grit blasting material used was angular SiC with the average size of 50 µm. The MAB treatment was conducted for 30 s to guarantee full coverage of the surfaces. The blasting pressure was 0.2, 0.4 and 0.6 MPa.

The deposition of the monolayer TiN coating was carried out in the arc plating PVD-unit PLATIT-π80, using the LAteral Rotating ARC-Cathodes (LARC) technology and standard deposition parameters. The coating deposition temperature was 450 °C. The thickness of coatings was 2.3 µm, and nanohardness 24 GPa. The WC-Co specimens (polished and cleaned in an ultrasonic bath with alcohol) were placed in a vacuum chamber after the cleaning procedure and mounted on the sample holder. Finally, samples were sputter-cleaned in argon plasma and a thin metallic Ti adhesion layer was deposited onto substrates prior to the coating (Fig. 1).

Post-treatment of the coating was used with micro-abrasive blasted as well grinded substrates. For post-treatment of the coating surface the OTEC® DF 35 polishing machine (drag finishing) was used. The samples were dragged in a circular motion at 25 m/min through a container, filled with grinding or polishing media. The H 1/400 walnut shell and the polishing paste P1, containing alumina grains, were used as grinding and polishing media, respectively; the time spent was 10 min for each treatment type.

Surface roughness (R_a and R_z) measurements of clean substrates, as-deposited and post-treated samples, were performed using a MAHR Perthometer. Results of measurements are shown in Tables 1 and 2. Each sample was measured in 6 different places to determine the average value.

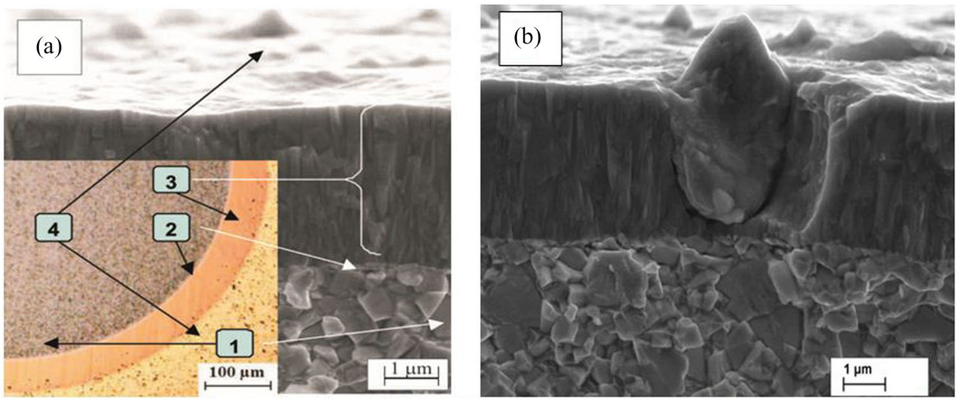

Fig. 1. (a) SEM image of the fractured surface of a TiN coating: (a) 1 – substrate; 2 – Ti adhesion layer; 3 – TiN layer; 4 – coating surface with macro-droplets; (b) cross-section of the coating with macro-droplets.

Table 1. Substrate and PVD coating surface roughness, µm

Sample identification	Substrate WC-Co		TiN as deposited		TiN+OTEC treated	
	R_a	R_z	R_a	R_z	R_a	R_z
R_a 0.05	0.06±0.01	0.50±0.09	0.09±0.02	1.16±0.55	0.07±0.02	0.88±0.29
R_a 0.20	0.19±0.03	1.66±0.35	0.23±0.03	2.43±0.80	0.24±0.04	2.34±0.84
R_a 0.40	0.45±0.03	3.37±0.34	0.48±0.05	3.76±0.96	0.46±0.08	3.51±0.44
R_a 0.80	1.04±0.12	6.52±0.77	1.02±0.16	6.56±0.59	1.04±0.14	6.57±0.83

Table 2. The effect of substrate pre-treatment pressure and coating post-treatment on surface roughness, µm (sample R_a 0.05)

Pre-treatment pressure, MPa	Substrate WC-Co		TiN as deposited		TiN+OTEC treated	
	R_a	R_z	R_a	R_z	R_a	R_z
0.2	0.37±0.03	3.00±0.62	0.37±0.05	2.99±0.83	0.35±0.03	2.60±0.37
0.4	0.58±0.07	4.42±0.77	0.60±0.05	4.47±0.81	0.58±0.05	4.10±0.46
0.6	0.70±0.03	5.08±0.63	0.70±0.07	5.10±0.65	0.68±0.08	4.65±0.81

For the determination of the adhesion of coatings, an experimental procedure, based on the CEN/TS 1071-8:2004 adhesion, test was used. Adhesion test was based on the Rockwell C hardness test using a conical diamond indenter and a 1471 N (150 kgf) indentation load. According to CEN/TS 1071-8, adhesion is divided into 4 categories: class 0 (high quality), class 1, class 2 and class 3 (poor quality). Class 0 indicates that no cracks or adhesive delamination is observed in the indent region and class 3 represents complete adhesive delamination of the coating. The indents of the adhesion test were examined by SEM.

Sliding wear study was performed at the ball-on-disk tester – CSM tribometer (normal load 1 N, linear speed 4.0 cm/s, distance 25 m, Ø6 mm Al_2O_3 ball) at the

Institute of Materials Research of the Slovak Academy of Sciences. The coefficient of friction and wear rate of coatings were determined. The sliding velocity and the distance were chosen to initiate wear in the coating and to measure wear rate of the coating. Tests were conducted at three different diameters (6, 8 and 10 mm) of one sample of each type of the substrate. Alumina balls were used as counter body to diminish the tribochemical wear. The laboratory tests were conducted at air and room temperature. The relative humidity was in the range of 35%–40%.

3. RESULTS AND DISCUSSION

3.1. Coating adhesion

Coating adhesion is mainly affected by the pre-treatment of the substrate surface. PVD coating adhesion, estimated by Rockwell hardness test indents after grinding and after micro-abrasive blasting (MAB), is the same in the case of grinded samples not standing high difference in roughness. The adhesion test results with a sample, pre-treated at 0.2 Mpa, are indicated in Fig. 2. Adhesion of grinded surfaces was classified as good (class 1). The MAB treatment results in inferior adhesion of the coating. Coating adhesion on MAB treated surfaces was classified as satisfactory (class 2).

3.2. Surface roughness

Using micro-abrasive blasting with SiC particles at different pressure of the polished sample with identification “R_a 0.05” (R_a 0.06 µm) it was demonstrated that surface roughness was increased as a result of higher air pressure up to R_a 0.70 µm or R_z 5.10 µm, at the pressure of 0.6 MPa (Table 2). Deposition of

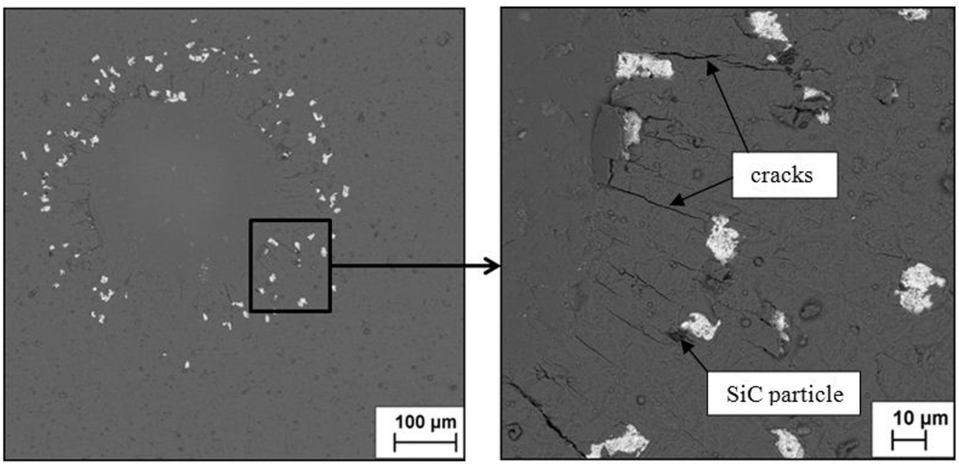

Fig. 2. SEM images of indents of adhesion test of coating (pre-treated 0.2 MPa).

the coating increased surface roughness R_a and R_z on grinded samples “R_a 0.05” and “R_a 0.20” up to 10%. Increasing of R_z can be explained by macro-droplets on the coating surface (Fig. 1b). On other samples (with higher roughness) macro-droplets have no significant influence on the surface roughness. In the result of drag grinding as post-treatment, the surface roughness reduces slightly due to the removing of higher peaks and macro-droplets on the surface.

Micro-abrasive blasting causes presumable contamination and residual stresses on the substrate surface. The SiC particles are sticked to the surface of the substrate and are unremovable during subsequent cleaning (Fig. 2). MAB is recommended as pre-treatment after the EDM process, where grinding can not be used. For grinding of tools MAB should be preferred to PVD to assure better adhesion.

3.3. Tribological properties

The roughness and CoF at sliding wear of the deposited as well as of the post-treated coating are indicated in Fig. 3. Post-treatment with selected parameters does not affect the surface roughness (Tables 1 and 2), except samples with low roughness. The influence of the post-treatment on the surface layer is minimal and only flattens the surface; therefore it has no effect on samples with high roughness. As follows from the above mentioned, post-treatment of the coatings is to be used to minimize the running in period and to reduce the CoF in dry sliding applications.

The influence of the surface roughness to the CoF is notable in case of grinded specimens, but not in case of MAB specimens. The MAB specimens have differences in the surface profile. Observations with SEM indicate that post-treatment removes the form surface macro-droplets and flattens the surfaces (Fig. 4).

Positive effect of the pre-treatment by grinding is expressed in the reducing of the CoF, caused by the flattening of the surface (removing of roughness peaks of the surface and macro-droplets on the coating). The higher the roughness, the higher is also CoF (Fig. 3).

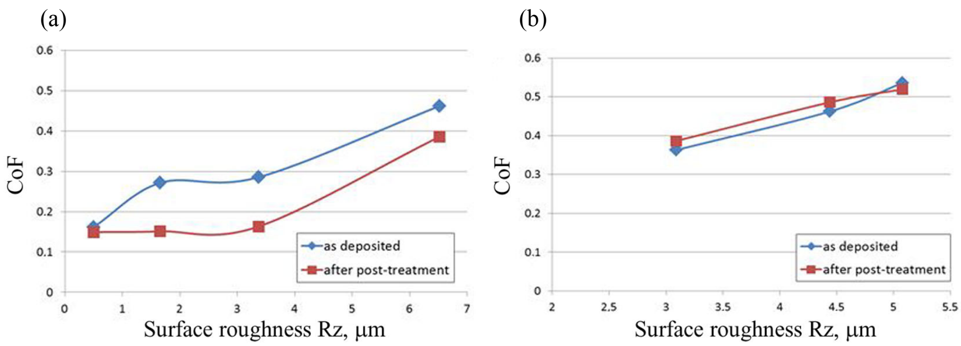

Fig. 3. The influence of the post-treatment of the TiN coating on the CoF: (a) grinded surface; (b) micro-blasted surface.

Wear mechanism of samples with different surface roughness, including high surface roughness, was studied. From the study of the wear tracks, indicated in Fig. 5, it follows that the highest peaks on the coating surface start to wear off at

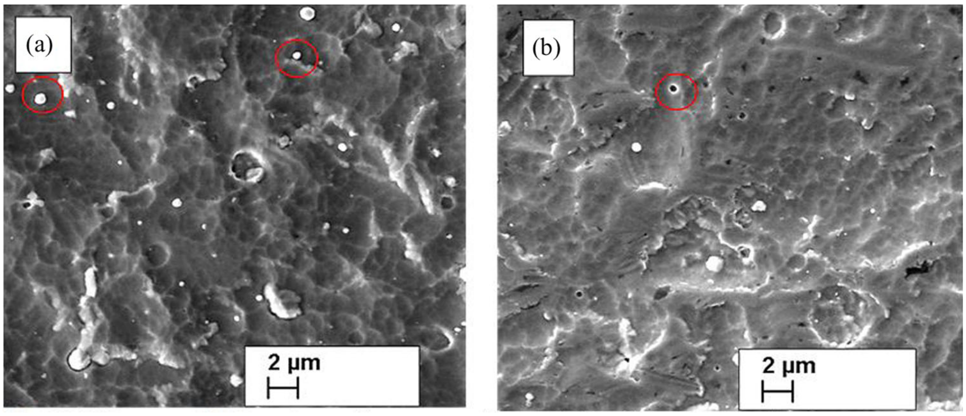

Fig. 4. SEM images of TiN coating surfaces: (a) as deposited; (b) post-treated.

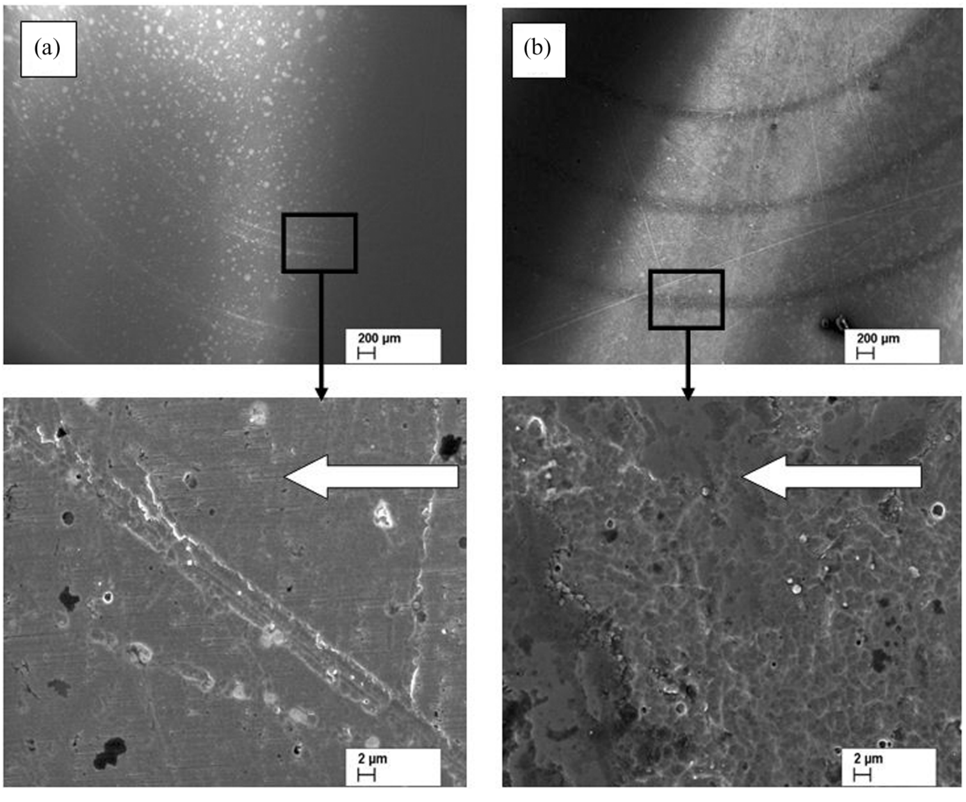

Fig. 5. SEM images of wear tracks of TiN after post-treatment: (a) R_a 0.05; (b) R_a 0.20 (arrow shows sliding direction).

the beginning of the test (Fig. 5b) and therefore the coating roughness decreases. Wear debris, formed during the test, were carried to grooves of the surface and actual wear rate was minimal.

4. CONCLUSIONS

1. Adhesion of the PVD coating on the WC-Co substrate depends on the pre-treatment of the substrate: grinded surfaces show better coating adhesion than micro-abrasive blasted surfaces and coating adhesion is lower with higher substrate surface roughness, especially by micro-blasted surfaces.
2. Tribological properties (e.g. CoF) are influenced mainly by the surface roughness, formed in the result of substrate pre-treatment and coatings post-treatment:
   - higher roughness of the coating surface causes the increase of the CoF; micro-abrasive blasted specimens show higher CoF than grinded surfaces with the same surface roughnesses;
   - post-treatment of the coating decreases the CoF in case of grinded surfaces and does not affect it in case of MAB pre-treated surfaces; post-treatment by grinding assures better adhesion and lower CoF compared with micro-abrasive blasting; the flattening of the surface takes place at the beginning of the test – highest peaks on the coating surface start to wear off.
3. The grinding of tools should be preferred to MAB to assure better adhesion.

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Pinna eel- ja pinde järeltöötluse mõju TiN-pinde omadustele

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Tänapäeval on füüsikalise aurustussadestuse pinded leidnud laialdast kasutamist, vähendamaks tööriistade kulumist. Õhukestel kõvapinnetel on vajalik kulumiskindlus ainult hea adhesiooni ja kindla pinnakareduse korral. Seepärast on vaadeldavas uurimistöös rakendatud kuivmikroabrasiivtöötlust SiC-abrasiiviga WC-Co alusmaterjalide eeltöötlusel. Järeltöötlust teostati, kasutades abrasiivis lihvimist (*drag grinding*). Vaadeldi pinde eel- ja järeltöötluse mõju TiN-kõvapinde adhesioonile ning triboloogilistele omadustele. TiN-kõvapinne paksusega 2,3 µm sadestati kaarleekaurustussadestusmeetodil. Pinnakaredust mõõdeti pinnakaredusmõõturi Mahr abil ja standardne Rockwelli adhesiooni katse viidi läbi, hindamaks TiN-pinde adhesiooni. Triboloogilised omadused tehti kindlaks, kasutades "kuul kettal" (*ball-on-disk*) meetodit. Eksperimentaalselt uuriti mikroabrasiivtöötluse mõju pinde triboloogilistele omadustele. Tuvastati seos pinde järeltöötluse ja hõõrdeteguri vahel.