

INFLUENCE OF THE SURFACE ROUGHNESS ON ADHESION OF CHROME COATINGS ON ALLOY TOOL STEEL X165CrMoV12

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ABSTRACT

The influence of surface roughness on the coating adhesion of electrodeposited chrome coatings with different roughnesses, has been evaluated using scratch testing technique. Coatings were deposited on the high alloyed tool steel. Photographs of the scratch grooves were recorded by the built-in optical microscope, at moments when critical loads had been reached. Crack pattern development was investigated. Results indicated that the increase of surface roughness lowers coating adhesion. Rough surfaces promote early coating failure, extensive spallation and early exposure of the substrate material.

Keywords: electrodeposited chrome coatings, scratch test, coating surface roughness.

AIMS AND BACKGROUND

Ever since the 1940's, chrome has been used to add a protective coating and shiny luster to a wide range of metal products, from home accessories to auto parts. Chrome plating is applied for wear resistance, lubricity, oil retention and other purposes. It can increase corrosion resistance, surface hardness, wear resistance and reduce friction^{1,2}. Chrome plating is easily applied and has a low cost³⁻⁶. Much progress has been realised during the last years in the field of coating deposition and improved coatings with excellent frictional properties^{1,7-10}. Chromium plating provides excellent hardness, bright appearance and resistance to corrosive environments. Problems such as matt deposition, milky white chromium deposition, rough or sandy chromium deposition, insufficient thickness and hardness are the most common problems faced in the electroplating industry¹.

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Deep drawing forming tools are widely used in the automotive industry. These tools are subjected to a wide range of load conditions depending on the part geometry. It is very important to achieve their long durability since they belong to a group of extremely costly parts. The tribological factors influencing the process of cold plastic forming are very important. Numbers of parameters are important when coating deposition is considered. In general, the surface topography has a great influence on the tribological behaviour^{11–13}. Novel techniques offer various new approaches to study physical, mechanical, structural and tribological properties of different chrome coatings^{5,6,9,14}. One of the efficient tools for tribological characterisation of coatings on nano- and micro-scale is scratch testing^{14–17}. It is specially suited technique to characterise practical adhesion failure of thin films and coatings.

The aim of this study is to investigate the influence of the coating surface roughness on coating adhesion of commercially available chrome plated high alloyed tool steel, using scratch test technique. Crack pattern development has been studied by means of microscopy techniques.

EXPERIMENTAL

Substrate material. For experimental investigations presented in this paper, domestic manufactured, thermally treated high alloyed tool steel with high toughness and hardness, marked with C4750 according to JUS standard (DIN 17006 designation: X165CrMoV12) has been selected, as a substrate material. Chemical composition of the C4750 steel is: 1.65% C, 0.30% Si, 0.30% Mn, max. 0.035% P, max. 0.035% S, 12.0% Cr, max. 0.25% Ni, 0.60% Mo, 0.10% V, 0.50% W. This steel belongs to a group of highly wear-resistant steels, due to the high volume of carbides in the microstructure, and is foreseen for cold work. The high carbon and chromium content promotes deep hardening. Hardenability is accentuated by small amounts of tungsten and molybdenum. Dimensional change in hardening is extremely low. Typical uses are long run blanking, stamping, cold forming dies; lamination dies; thread rolling dies; trimmer dies; slitters; lathe centers; brick mold liners; gages; cutting and punching tools and abrasion resistant liners⁵.

Sample plates with 70 mm × 40 mm × 5 mm dimensions were manufactured for testing purposes. Before final machining by abrading, quenching in oil and stress relieving were performed with resulting hardness of 60–63 HRC. Prior to deposition, stainless steel samples were grinded in order to obtain different surface roughness. Different surface roughness was achieved simply by using different grain sizes in a wet grinding process. 4 different groups of steel samples were produced and then deposited with chrome coating. In this way 4 groups of samples with 4 different coating surface roughnesses were produced for testing purposes.

Coatings. Hard chrome plated steel samples were produced by electrodeposition technique. Thickness of the chrome coating before final machining was approximately 0.2 mm and after final abrading and polishing, was in range 5–50 μm . Surface defects were not noticed after coating deposition. The following values of coating surface roughness were obtained: $R_a = 0.01 \mu\text{m}$ (denoted by Cr0.01 further in the text); $R_a = 0.08 \mu\text{m}$ (denoted by Cr0.08 further in the text); $R_a = 0.4 \mu\text{m}$ (denoted by Cr0.4 further in the text); $R_a = 0.5 \mu\text{m}$ (denoted by Cr0.5 further in the text).

Scratch testing. A controlled scratch with a diamond tip was realised, under linear progressive load, using CSM scratch tester instrument. At some critical load the coating starts to fail. These critical loads are detected very precisely by means of an acoustic sensor attached to the load arm and recorded by the tester. Also, optical photograph of the scratch groove at the moment when the critical load is achieved is automatically recorded by the built in optical microscope. The tester also monitors and records the normal load, the friction force and the penetration depth.

Scratch tests were carried out with the following preferences: linear progressive type scratch; maximum load of 100 N; loading rate of 100 N/min and speed of 10.05 mm/min. Schematic presentation of the scratch test is given in Fig. 1a. Rockwell diamond stylus indenter was used with tip radius of 50 μm , as shown in Fig. 1b. It is a procedure compatible with ASTM D7187 standard. Sliding direction was set perpendicular to the orientation of final grinding of the coating surface. 3 scratch experiments were performed for all materials and all were repeated 3 times. All experiments were performed under dry sliding conditions in controlled room temperature of 20–22 $^{\circ}\text{C}$ and relative humidity of 35–40%. Prior to testing, each sample was thoroughly cleaned by alcohol, then cleaned in ultrasonic bath for 60 min and dried in hot air afterwards.

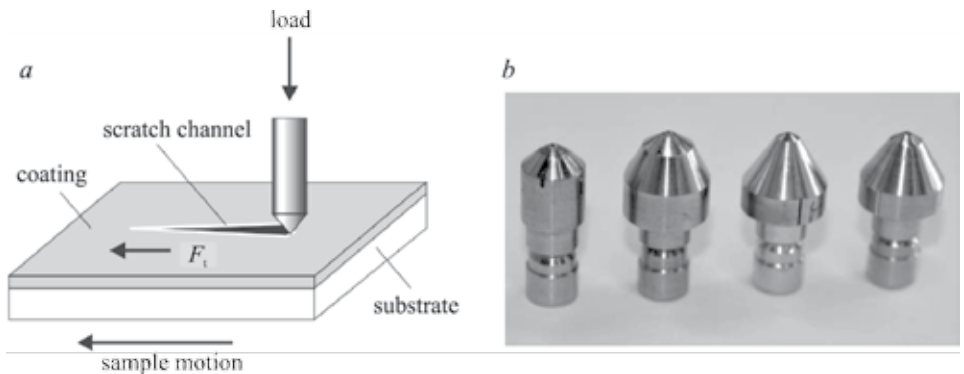


Fig. 1. Scratch test (a) and rockwell diamond stylus indenter¹⁸ (b)

RESULTS AND DISCUSSION

The scratch test is commonly used for evaluation of the adhesive and cohesive strength of films. Only some of the observed failure events in scratch testing are related to detachment at the film–substrate interface and are thus relevant as a measure of adhesion. The load at which a specific failure event is recorded is called the critical load (L_c). The smallest load, L_{c1} , is normally the first critical failure point and corresponds to the onset of cracking failure. Load L_{c2} usually indicates film spallation or detachment, crack opening and growth, and load L_{c3} indicates total delamination of the coating. Critical failure modes during scratch

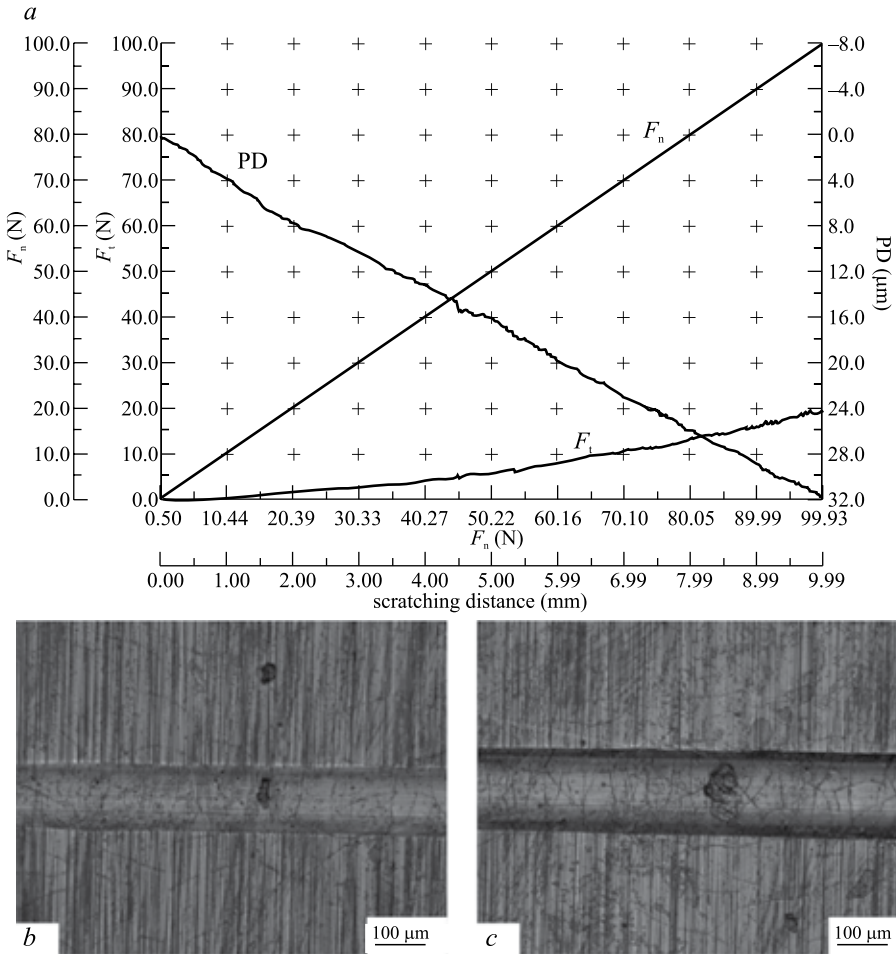


Fig. 2. Diagram of the friction force, normal force and penetration depth during scratch testing of the chrome coating (*a*), $R_a = 0.01 \mu\text{m}$, optical micrographs of the scratching scar at critical loads: L_{c1} (*b*) and L_{c2} (*c*)

testing are subject to many ongoing investigations^{19,20}. Sliding of the diamond stylus over coating surface can cause one or several failure modes such as: adhesive rupture, surface buckling, adhesion/cohesion, forward cracking, rearward cracking or else, depending on many influential factors (substrate/coating materials features, environment characteristics, testing parameters, different phases of testing and other).

Real time diagrams of the friction force, normal force and penetration depth during scratch testing for observed samples are shown in Figs 2–5. Also, the typical images of the scratch track recorded by optical microscopy, at moments when critical loads are achieved, are shown in Figs 2–5.

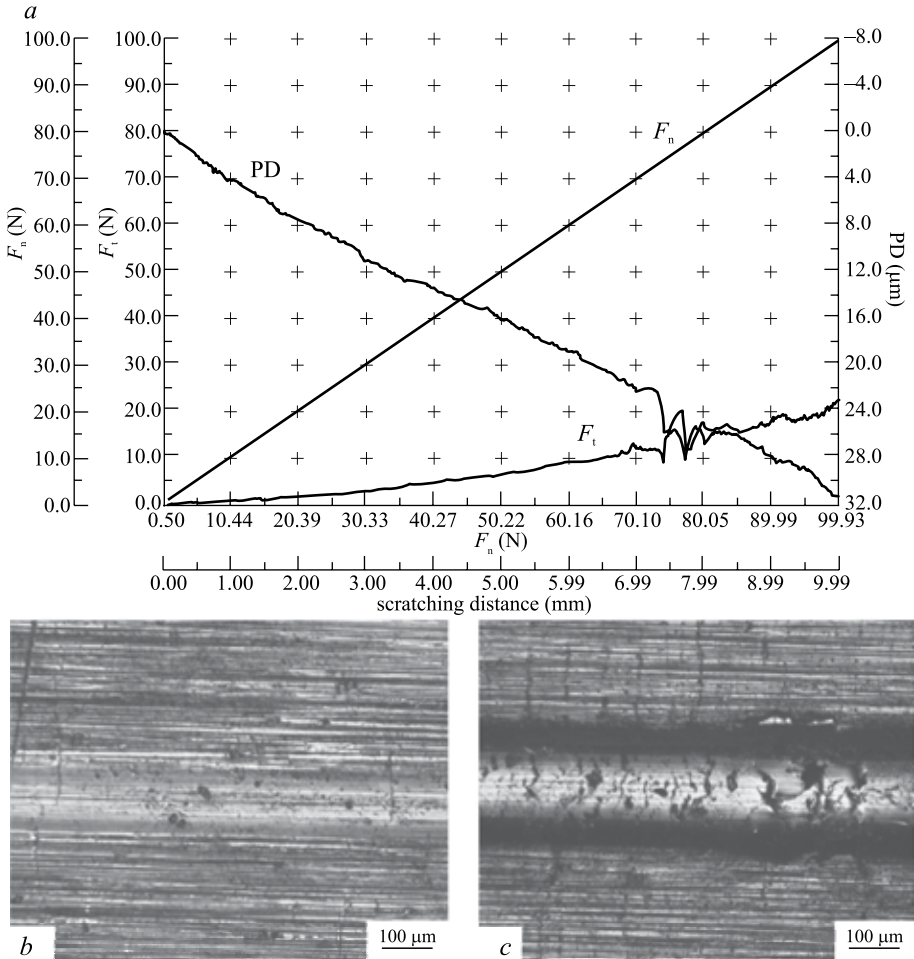


Fig. 3. Diagram of the friction force, normal force and penetration depth during scratch testing the chrome coating (a), $R_a = 0.08 \mu\text{m}$, optical micrographs of the scratching scar at critical loads: Lc_1 (b) and Lc_2 (c)

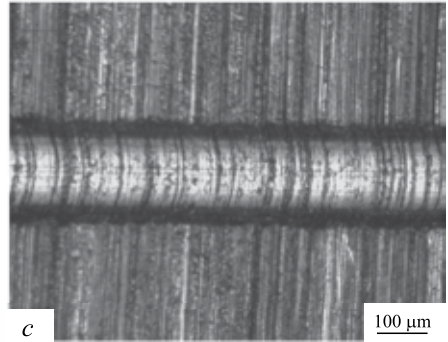
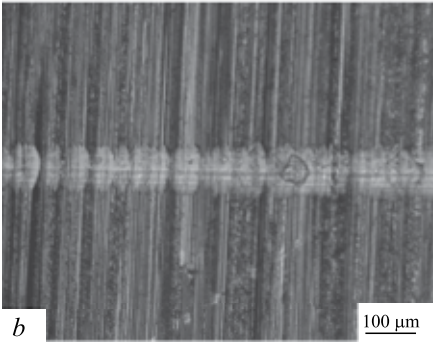
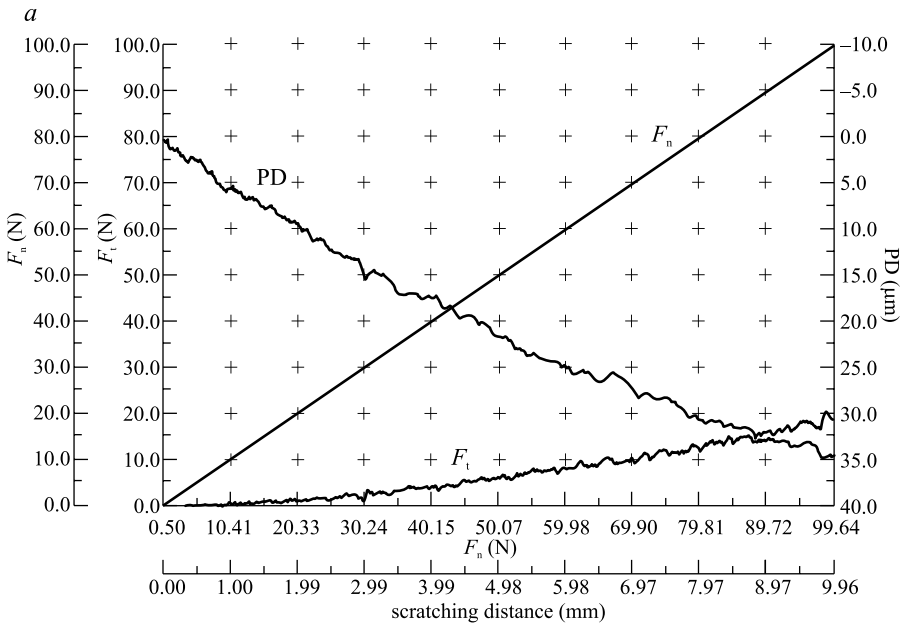


Fig. 4. Diagram of the friction force, normal force and penetration depth during scratch testing the chrome coating (*a*), $R_a = 0.4 \mu\text{m}$, optical micrographs of the scratching scar at critical loads: L_{c1} (*b*) and L_{c2} (*c*)

It can be considered that chrome coating with $R_a = 0.01$ surface roughness has rather good adhesion properties, because substrate material on the bottom of the track is only visible when the critical load L_{c2} is achieved and there was no extensive delamination. Coating failure appears as a network of fine thin cracks and at L_{c2} load, there was a single round-shape spallation (total delamination) observed. The friction force curve had no significant oscillations. The behaviour of the chrome coating with $R_a = 0.08 \mu\text{m}$ surface roughness was similar to the previous one, but slightly higher roughness produced larger cracks and significant change of the friction force in the later period of scratching. It is probably the con-

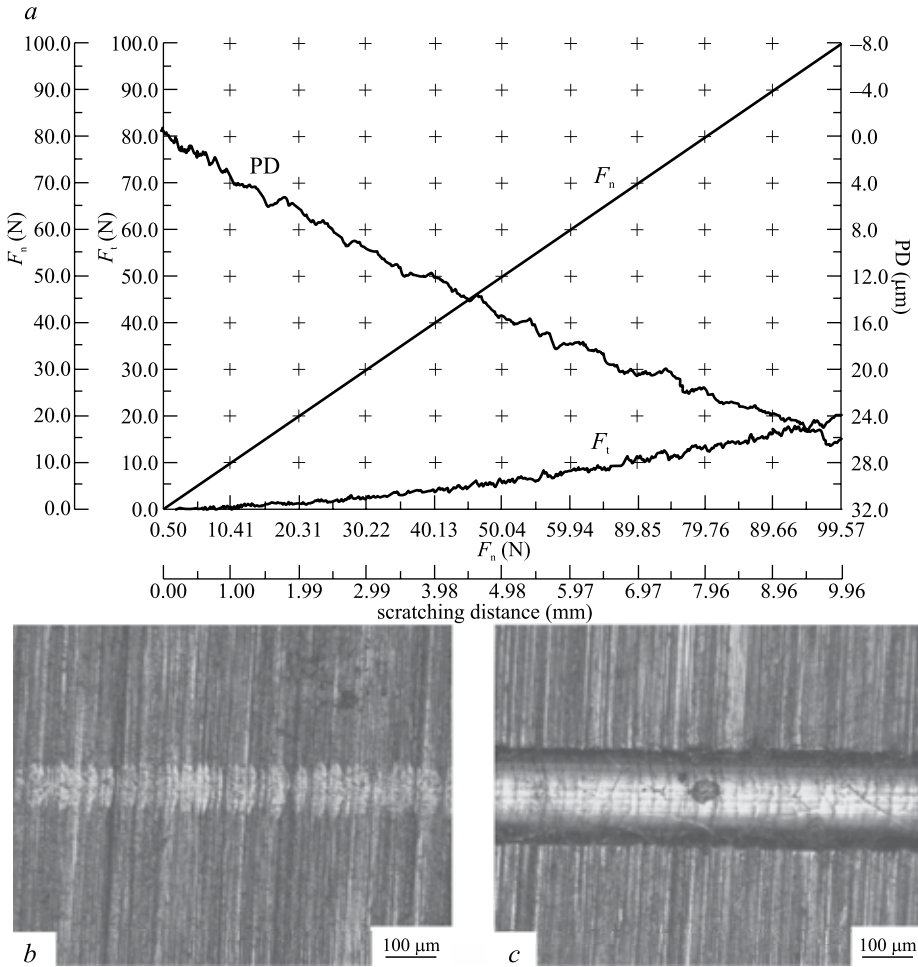


Fig. 5. Diagram of the friction force, normal force and penetration depth during scratch testing the chrome coating (a), $R_a = 0.5 \mu\text{m}$; optical micrographs of the scratching scar at critical loads: L_{c1} (b) and L_{c2} (c)

sequence of the large asperity contact with the indenter tip which produced asperity fracture and abrupt change of the friction force due to adhesion and fracture. Rough samples ($R_a = 0.4 \mu\text{m}$; $R_a = 0.5 \mu\text{m}$) produced many cracks already at the first critical load, followed by extensive delamination of the coating during later phases of testing (Figs 4 and 5).

The complex surface contact processes (contact deformation and adhesion) have significant effects on the friction force. As the 2 surfaces come into contact, surface roughness and asperities are deformed into a smooth, polished intermediate film^{5,6}. The higher roughness provides less conformal contact and at the

same time it provokes easier fracture of asperities. In case of smooth surfaces, plastic deformation is more probable to appear and adhesion between materials in contact plays significant role. Large asperities provoke early spallation of the coating what can be clearly observed in Fig. 4b (round chipping of the coating in the central region of the scratch groove).

It is obvious that fine and rough surfaces produce different mechanisms of coating failure. In the case of smooth surfaces, higher adhesion is achieved, coating cracking is in a form of a transverse semi-circular cracks in the central region of the scratch groove. Coating spalling and coating breakthrough occurred at higher normal loads. In the case of rough coating surface, more brittle behaviour occurred. Appearance of the cracks pattern produced after scratching on the chrome coating of $R_a = 0.5 \mu\text{m}$ surface roughness is shown in Fig. 6. Scratch groove has many irregular large crevices and cracks, coating spalling and chipping. Also, exposed substrate is earlier visible.

Comparison of the critical loads obtained by the tester during the scratch test is given in Fig. 7. It can be clearly noticed that comparison of both critical loads

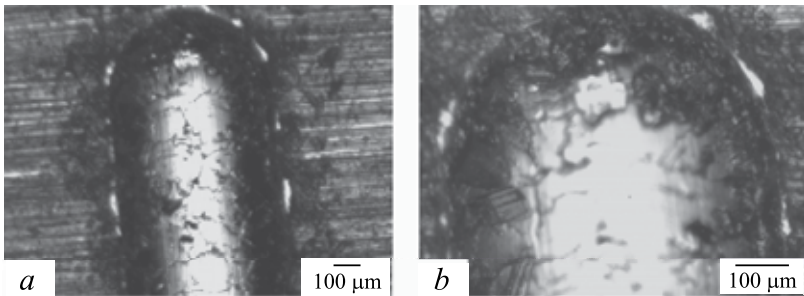


Fig. 6. Crack pattern produced after scratching, on the chrome coating of $R_a = 0.5 \mu\text{m}$ surface roughness: the scratch groove showing the area surrounding the track (a); higher magnification of the scratch groove showing coating spalling, crevices and cracks (b)

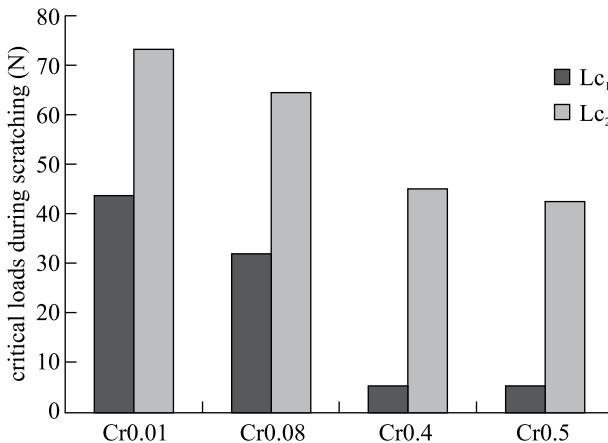


Fig. 7. Influence of the coating surface roughness on coating adhesion: critical loads during the scratch test for different coating surface roughness

(Lc_1 , Lc_2) indicates better quality of chrome coatings with fine roughness. In case of coatings with fine surface roughness, critical load, Lc_1 was almost 5 times of the rough coatings, indicating significantly better adhesion.

The most desirable properties of the chromium as a metal coating are its inherent protective and decorative characteristics. Many performance criteria have been imposed on chrome coatings used in industry and scratch resistance is among important requirements. This is especially true in automotive industry where coated forming tools are present. Scratch resistance is also required in those applications where appearance of the coated surface is important, such as decorative chromium plating in base metal protection. Scratch testing enables quantification of adhesive strength for coating quality validation thus representing efficient tribological tool for research, development and quality control.

CONCLUSIONS

It can be considered that chrome coating with $R_a = 0.01$ surface roughness has rather good adhesion properties. In case of coatings with fine surface roughness, the first critical load was almost 5 times of the rough coatings, indicating significantly better adhesion. Analysis of optical photographs of the scratch groove indicated different mechanisms of crack pattern development for smooth and rough surfaces. Large crevices and cracks and delaminated areas of coating can be observed on rough surfaces. Also, exposed substrate is earlier visible.

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REFERENCES

1. M. BAYRAMOGLU, B. ONAT, N. GEREN: Statistical Optimization of Process Parameters to Obtain Maximum Thickness and Brightness in Chromium Plating. *J. of Materials Processing Technology*, **203**, 277 (2008).
2. K. R. NEWBY: Functional Chromium Plating. *Metal Finishing*, **105** (10), 182 (2007).
3. K. DOHDA: Tribological Properties of Thin Hard Coatings Used in Metal Forming. In: *Proc. of the Japan International Tribology Conference, Nagoya, 1990, 1973–1980*.
4. A. DARBEIDA, J. von STEBUT, M. BARTHOLE, P. BELLIARD, L. LEAIT, G. ZACHARIE: Comparative Tribological Study of Chromium Coatings with Different Specific Hardness. *Surface and Coatings Technology*, **68/69**, 582 (1994).
5. *ASM Handbook – Forming and Forging*. Vol. 14, ASM, 9th ed. *Metals Handbook*, ASM International Handbook Committee, 1988.

6. ASM Handbook – Surface Engineering. Vol. 5, ASM, 10 ed. Metals Handbook, ASM International Handbook Committee, 1994.
7. D. V. SHTANSKY, N. A. GLOUSHANKOVA, I. A. BASHKOVA, M. I. PETRZHNIK, A. N. SHEVEIKO, F. V. KIRYUKHANTSEV-KORNEEV, I. V. RESHETOV, A. S. GRIGORYAN, E. A. LEVASHOV: Multifunctional Biocompatible Nanostructured Coatings for Load-bearing Implants. *Surface and Coatings Technology*, **201**, 3732 (2006).
8. V. GOROKHOVSKY, C. BOWMAN, P. GANNON, D. van VOROUS, A. A. VOEVODIN, A. RUTKOWSKI, C. MURATORE, R. J. SMITH, A. KAYANI, D. GELLES, V. SHUTTHANANDAN, B. G. TRUSOV: Tribological Performance of Hybrid Filtered Arc-magnetron Coatings. Part I. Coating Deposition Process and Basic Coating Properties Characterization. *Surface and Coatings Technology*, **201**, 3732 (2006).
9. E. BEMPORAD, C. PECCHIO, S. de ROSSI, F. CARASSITI: Characterisation and Wear Properties of Industrially Produced Nanoscaled CrN/NbN Multilayer Coating. *Surface and Coatings Technology*, **188–189**, 319 (2004).
10. J. LIN, S. CARRERA, A. O. KUNRATH, D. ZHONG, S. MYERS, B. MISHRA, P. RIED, J. J. MOORE: Design Methodology for Optimized Die Coatings: The Case for Aluminum Pressure Die-casting. *Surface and Coatings Technology*, **201**, 2930 (2006).
11. B. PODGORNIK, S. HOGMAR, O. SANDBERG: Influence of Surface Roughness and Coating Type on the Galling Properties of Coated Forming Tool Steel. *Surface and Coatings Technology*, **184**, 338 (2004).
12. P. HARLIN, P. CARLSSON, U. BEXELL, M. OLSSON: Influence of Surface Roughness of PVD Coatings on Tribological Performance in Sliding Contacts. *Surface and Coatings Technology*, **201**, 4253 (2006).
13. P. HARLIN, U. BEXELL, M. OLSSON: Influence of Surface Topography of Arc-deposited TiN and Sputter-deposited WC/C Coatings on the Initial Material Transfer Tendency and Friction Characteristics under Dry Sliding Contact Conditions. *Surface and Coatings Technology*, **203**, 1748 (2009).
14. K. BEWILOGUA, G. BRAUER, A. DIETZ, J. GAEBLER, G. GOCH, B. KARPUSCHEWSKI, B. SZYSZKA: Surface Technology for Automotive Engineering. *CIRP Annals – Manufacturing Technology*, **58**, 608 (2009).
15. S. J. BULL: Can Scratch Testing Be Used as a Model for the Abrasive Wear of Hard Coatings? *Wear*, **233–235**, 412 (1999).
16. P. HEDENQVIST, S. HOGMARK: Experiences from Scratch Testing of Tribological PVD Coatings. *Tribology International*, **30**, 507 (1997).
17. J. von STEBUT: Multi-mode Scratch Testing – An European Standards, Measurements and Testing Study. *Surface and Coatings Technology*, **200**, 346 (2005).
18. The Influence of the Rockwell Indenter Tip Geometry on the Progressive Load Scratch Test. *Applications Bulletin, CSM Instruments*, **26** (2008).
19. P. J. BURNETT, D. S. RICKERBY: The Relationship between Hardness and Scratch Adhesion. *Thin Solid Films*, **154** (1–2), 403 (1987).
20. P. J. BURNETT, D. S. RICKERBY: The Scratch Adhesion Test: An Elastic-plastic Indentation Analysis. *Thin Solid Films*, **157** (2), 233 (1988).

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