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## SURFACE ROUGHNESS AND ITS EFFECT ON TRIBOLOGICAL PERFORMANCE OF MAGNETRON SPUTTERED NITRIDE COATINGS

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**Abstract:** Tribological performance of hard coatings greatly relies on surface roughness. Achieving an adequate performance is a counter play between desired coating properties and production costs, thus determination of optimal surface roughness is always a challenge. In order to investigate the influence of the surface roughness on coatings tribological behaviour, three kinds of nitride coatings (TiAlN, ni-AlTiN/TiN, TiAlN/CN<sub>x</sub>) were deposited by magnetron sputtering on substrates with different grades of surface roughness. Surface morphology before and after the coating process was characterized using stylus profilometry and atomic force microscopy (AFM). Coating mechanical properties and adhesion were assessed by nanoindentation and scratch test, respectively. Wear resistance was evaluated by tribological tests conducted in dry reciprocating sliding conditions using Al<sub>2</sub>O<sub>3</sub> as a counterpart material. It was found that the surface roughness of all investigated samples increased after the deposition process. This is mainly due to the formation of defects in the growing coating. It was found that the scratch adhesion and wear resistance of all tested coatings is deteriorated with drastically increased surface roughness. However, in a domain of low surface roughness ( $S_a = 40 - 100$  nm) changes in morphology of TiAlN and TiAlN/CN<sub>x</sub> coatings does not affect the coatings scratch adhesion nor wear rate. Also the friction coefficient of these coatings is not dependent on the surface roughness in this domain. Contrary, the nanolayered AlTiN/TiN coating displayed a sensitivity of the scratch adhesion and sliding wear on the surface roughness. Presented findings are beneficial in future design of optimal wear resistant coated systems.

**Keywords:** hard coating, magnetron sputtering, surface roughness, wear, reciprocating sliding, coefficient of friction.

### 1. INTRODUCTION

Application of PVD hard coatings in a various fields of mechanical engineering is an ever expanding area. Accordingly the development of new and improvement of existing wear resistant coating systems is still a technological challenge and a progressive scientific research field. Optimisation of a wear resistant coating design requires detailed knowledge about parameters involved not only

in the surface exploitation but also in manufacturing parameters of part to be coated, like initial surface roughness, machining processes applied in surface production etc.

Wear resistant PVD coatings are usually very hard and stiff materials [1] that in tribo-contact behave differently than conventional materials [2]. In such contacts, on a micro scale plastic deformation is limited, so the real contact between rough surfaces occurs at much smaller areas [2]. In this way heat input

at asperities is increased and the contact pressure raised, which induces local coating cracking and fragmentation. Such scenario leads to completely different behaviour of the tested system as compared to the same coating characterised by smooth surface [2,3].

The majority of tribological investigations concern coatings on very smooth surfaces where the roughness and coating defect effects are neglected [2,3]. Although the coating performance highly relies on its mechanical properties, results of such tests usually overestimate the performances, as compared to the same coatings applied on real rough surfaces.

Bearing in mind that machining processes used for production of surfaces with finest finish considerably increase the product overall cost, use of such operation should be minimised [3]. Accordingly, the coating performance should be tested over a range of surface roughness.

The aim of this study is to investigate the effect of surface roughness on tribological performance of three kinds of hard coatings that also represent different layer architectures. Namely, TiAlN which is a single layer, TiAlN/CN<sub>x</sub> which is a double layer and AlTiN/TiN which is a nanolayered coating. Investigated nitride coatings are produced by industrial magnetron sputtering systems.

## 2. EXPERIMENTAL PROCEDURES

Substrates for coating deposition were made of quenched 100Cr6 steel in a form of disk whose dimension were  $\varnothing 20 \times 5$  mm. Standard metallographic procedure was employed for surface preparation. Samples were prepared using 400, 1500, and 2000 grit silicon carbide sandpaper. The specimen 2000 was additionally polished by using a 3  $\mu\text{m}$  diamond polishing paste. Roughest surface was produced by conventional flat grinding procedure using regimes for rough grinding. Samples are denoted with the respect to the grit numbers: 400, 1500, 2000 and CG for the conventionally grinded surface.

Three kinds of coatings were deposited by two closed field unbalanced magnetron

sputtering systems equipped with four targets. TiAlN coating was deposited using CC800/7 (CemeCon) while nano-layered AlTiN/TiN ( $\sim 40$  nm period) and double layered CN<sub>x</sub>/TiAlN coating were deposited by CC800/9 (CemeCon) system. Coating thickness was determined by standard ball cratering method. The hardness of the coatings was acquired by nano indentation method employing a Vickers prism in H100C (Fischerscope) hardness tester. Hardness was determined from the indentations where the final indentation depth was one tenth of the coating thickness.

Surface topography before and after the deposition, as well as morphology of wear tracks were acquired by 3D-profilometer (Taylor Hobson Talysurf). Scans were taken on the areas of 1 mm<sup>2</sup> with high lateral and height resolution (2  $\mu\text{m}$  in *x* and *y* directions and 10 nm in *z* direction).

Scratch adhesion of the coated systems was evaluated by Revetest (CSM) device. Progressive loading test to a final value of 150 N was performed over the scratching distance of 4.5 mm. The test was conducted sliding a Rockwell indenter with a tip radius of 200  $\mu\text{m}$  with velocity of 6 mm/min. Critical forces, which lead to typical coating failures, were determined by monitoring acoustic signals and by optical examination of generated scratch tracks.

Dry reciprocating sliding tests were conducted in air by CSM standard tribo-tester. As a counterpart in all tests Al<sub>2</sub>O<sub>3</sub> ball with a 6 mm diameter was used. In these tests normal load of 5 N and sliding velocities of 5 cm/s were employed. Stroke length of 5 mm was chosen and test was interrupted after 2000 sliding cycles. During these tests coatings were not totally worn out.

Cross sectional measurements of the wear tracks conducted by profylometer were used for the calculation of specific wear rate. The specific wear rate *K* is calculated using the equation (1), where *V* is the wear volume [mm<sup>3</sup>], *F<sub>N</sub>* is the normal load [N], and *l* is the sliding distance [m].

$$K = \frac{V}{F_N l}. \quad (1)$$

### 3. RESULTS AND DISCUSSION

Investigated coatings are representatives of three kinds of coating designs. These are monolayer (TiAlN), double layer (CN<sub>x</sub>/TiAlN) and nano-layered (nl-AlTiN/TiN) coatings. Properties of investigated coatings are summarised in Table 1. Thickness of the investigated coatings is nearly the same, while the differences in hardness and  $H^3/E^{*2}$  ratio are considerable.

**Table 1.** Thickness and mechanical properties

|                 | TiAlN  | nl-AlTiN/TiN              | CN <sub>x</sub> /TiAlN                    |
|-----------------|--------|---------------------------|---|
| $H$ [GPa]       | 33.4   | 24.9                      | 15.6                                      |
| $E^*$           | 319    | 329                       | 257                                       |
| $H^3/E^{*2}$    | 0.37   | 0.14                      | 0.06                                      |
| Thickness [μm]  | 4.9    | 6.3                       | 6.3                                       |
| Layer thickness | 4.9 μm | 40 nm double layer period | CN <sub>x</sub> : 0.9 μm<br>TiAlN: 5.4 μm |

Roughness values of the sample surfaces before and after the deposition process are presented in Table 2. Comparing with surfaces from the real production, substrates roughness achieved in this investigation ( $S_a = 10 - 50$  nm) belongs to the domain of very fine surface finishes. Applying industrial machining processes for achieving such fine surface finishes induce a considerable increase of production costs. Therefore, it would be of great interest to reduce the required surface finish and still obtain the maximum of a coated system.

In most cases, the surface roughness increased after the deposition process (Table 2). 3D profylometer images presented in Figure 1 provide better insight into the changes caused by the deposition. Peaks

present on coated surfaces are nodular growth defects. Those growth defects are present in layers of all coatings investigated in this study [4]. Although, a magnetron sputtering system incorporates fewer defects than other deposition techniques, their presence is unavoidable in such industrial deposition unit.

Surface roughness after the coating process converged to some value that is dependent on the coating type. However, the roughest samples (CG) displayed the least changes in surface morphology what is explained by its initial grooves and ridges which are higher than the sizes of growth defects.

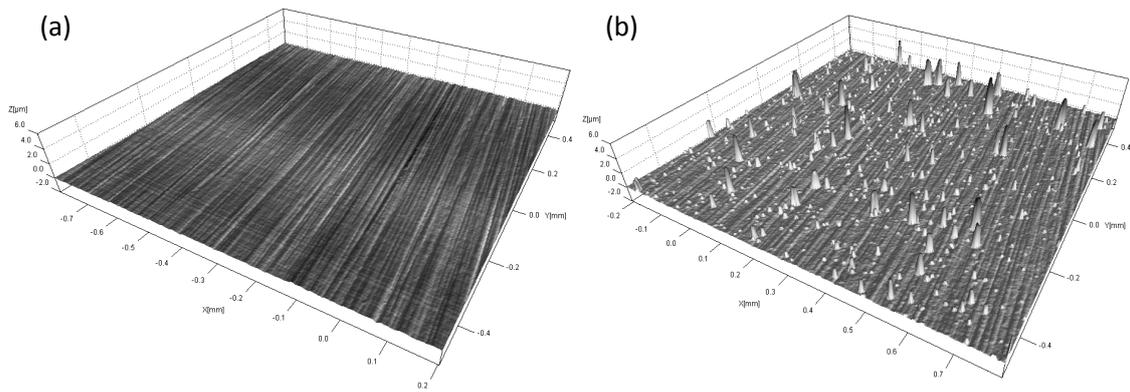
All tested coatings display very high scratch adhesion and mostly fail by ductile modes, except the CN<sub>x</sub>/TiAlN coating, see Fig. 2. For this coating, a wedging spallation is observed [5] that is a consequence of strong coating and a weaker interface. In addition, a low  $H^3/E^{*2}$  ratio indicates that this coating is unable to accommodate the extensive deformations of the underlying substrate [1].

Analysis of the scratch tracks revealed that coating scratch adhesion, in the domain of smooth surfaces, is mostly independent of the surface roughness. For particular coating, typical coating failures occurred almost at the same loads, usually in the range of the test deviations. The scratch test results are presented in form of the diagram in Figure 3. It is common for all tested coatings that the roughest CG sample exhibited the weakest scratch adhesion.

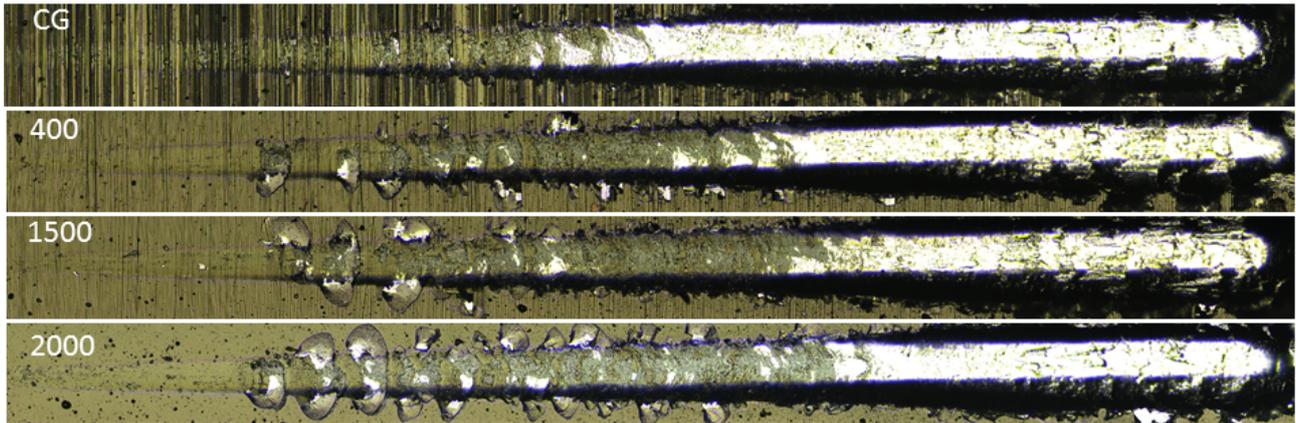
Adhesion of TiAlN and CN<sub>x</sub>/TiAlN in a domain of a low surface roughness is characterised by a negligible dependence on the surface roughness. However, the nl-AlTiN/TiN coating has the highest scratch adhesion and it is the most sensitive to the

**Table 2.** Average surface roughness  $S_a$  [nm] of the investigated surfaces

| Sample | TiAlN  |       | nl-AlTiN/TiN |       | CN <sub>x</sub> /TiAlN |       |
|--------|--------|-------|--------------|-------|------------------------|-------|
|        | Before | After | Before       | After | Before                 | After |
| 2000   | 11.0   | 71.3  | 10.5         | 39.1  | 10.6                   | 65.2  |
| 1500   | 19.3   | 58.2  | 14.9         | 40.2  | 18.0                   | 108.7 |
| 400    | 48.5   | 64.8  | 41.7         | 97.7  | 46.1                   | 99.8  |
| CG     | 525.7  | 571.4 | 545.1        | 545.9 | 467.0                  | 493.2 |



**Figure 1.** Surface morphology of the nl-AlTiN/TiN sample: (a) before and (b) after the deposition



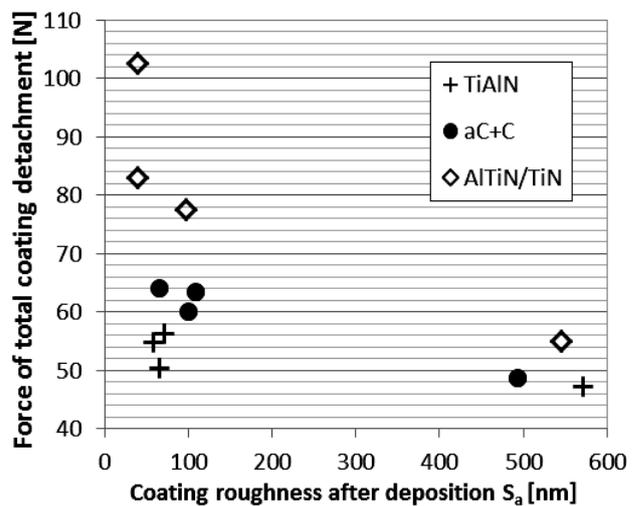
**Figure 2.** Representative scratch tracks on the surfaces with different roughness,  $CN_x/TiAlN$  coating

roughness change. This highest adhesion is achieved for the 1500 sample, while only small difference is found when comparing sample 2000 and sample 400. This finding suggest that the behaviour of nl-AlTiN/TiN coating conforms with findings of our previous study [6] that the highest coating adhesion is not necessarily achieved at the smoothest surface possible. The fact that the 2000 and 1500 samples of nl-AlTiN/TiN behave differently suggest that, depending on the substrate morphology, nanolayered coating display specific response to the shearing stresses.

Observations of the scratch tracks of nanolayered coating indicate that this coating fails with fewer cracking than the other two. This is a consequence of nanolayered design that under load promotes step-like cracking and prevents coating column shearing and bending [7].

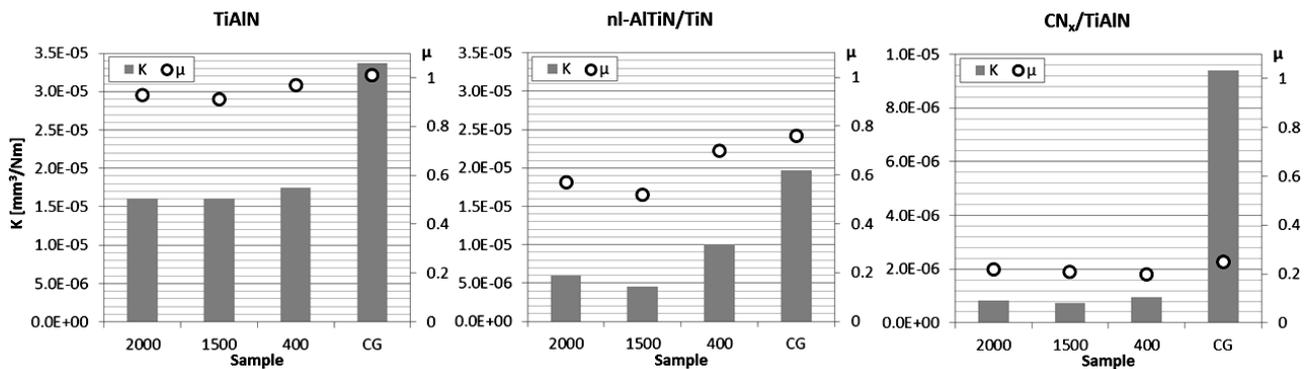
$CN_x/TiAlN$  coating displayed lower critical forces than the nl-AlTiN/TiN while still higher than the TiAlN coating. These increased critical forces over the TiAlN coating are addressed to lower coefficient of friction (COF) that

introduce lower shear stresses and prolongs the coating failure.



**Figure 3.** Critical force of the coating total detachment ( $L_{c3}$ ) in function of the surface roughness

The results of tribological tests are presented in form of coefficient of friction (COF) and specific wear rate ( $K$ ) in Figure 4. COF of the TiAlN coating has very high values (0.95) while conversely the  $CN_x/TiAlN$  exhibits very low values (0.2), typical for the carbon



**Figure 4.** Coefficient of friction ( $\mu$ ) and specific wear rate ( $K$ ) of various coating systems

coatings. In the tested range of surface roughness both TiAlN and CN<sub>x</sub>/TiAlN coatings show no dependence of COF on the surface roughness. Additionally, in a domain of low surface roughness wear rate of the coating is independent of the surface roughness. Obviously, both coatings display no sensitivity to the ploughing wear caused by asperity interactions. For these coatings increased wear is detected only for the roughest sample.

Regarding the performance of ni-ALTiN/TiN coating in tribological tests, it is placed in between the performances of the other two tested coatings. Interestingly the friction coefficient of the ni-ALTiN/TiN coating is found to be dependent on the surface roughness to some extent. This was found for two roughest samples tested, the 400 and CG sample. Additionally, for these two samples increase in wear rate was detected and it correlates with increased coefficient of friction. Such finding suggests that sliding in these contacts is hampered due to increased ploughing or extensive wear debris formation.

Comparison of the coating performances indicates that absolute amounts of the coating wear correlates well with the values of friction coefficient. Additionally, the wear of a counter ball correlates with wear of a particular coating tested.

Increased wear of the coatings with rough surfaces in tribo-contact is linked to the raised local contact pressure at the asperities [2,6]. High contact pressure induces cracking, local coating fragmentation and introduction of these fragments into the tribo-contact [2]. These, all together with fragments of nodular defects, and counter material, substantially

increase the abrasion and overall wear. Therefore, this dramatic increase in coating wear rate should be addressed to the change of dominant wear mechanism. In a domain of low surface roughness mild abrasion and oxidation are the most pronounced wear mechanisms. By increase of surface roughness, as a consequence of pressure and surface fatigue, flaking and fragmentation prevails. Apparently, this mechanism exchange occurs somewhat earlier for the ni-ALTiN/TiN coating. Presented findings suggest that, when applying a nanolayered coating, during design of mechanical parts special attention should be given to the surface morphology.

#### 4. CONCLUSIONS

PVD coatings examined in presented investigation differ in chemical composition, architecture, mechanical and tribological properties. Therefore, these coatings manifested different performances with roughness variation. The main conclusions are listed in the following:

- Due to a nodular coating defect formation, the deposition process induced a considerable change of the initial surface morphology of all samples. Smooth surfaces with different initial roughness tended to approximately the same roughness ( $S_a$ ) after the coating deposition.
- In a domain of low surface roughness, only the nanolayered ALTiN/TiN coating displayed a sensitivity of the coating scratch adhesion on the surface roughness.

- For all coatings the coarse grinded surfaces exhibited the lowest values of scratch adhesion and a high wear rate in dry reciprocating sliding test.
- In sliding tribo-tests friction coefficient and wear of TiAlN and CN<sub>x</sub>/TiAlN coatings in a domain of low surface roughness are not affected by surface roughness.
- Only the nanolayered AlTiN/TiN coating showed dependence of the friction coefficient and wear on the surface roughness. It is suggested that, nanolayered AlTiN/TiN coating is prone to be worn by surface fatigue and flaking that become dominant wear mechanisms with increase in surface roughness.
- Achieving the highest adhesion and wear resistance of coated systems is feasible if the substrate preparation includes fine surface treatments after the coarse grinding process. However, the best coating performance is already attainable on surfaces with roughness about S<sub>a</sub> ~ 40 nm, which are higher than those produced by finest polishing treatments.

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## REFERENCES

- [1] A. Leyland, A. Matthews: On the significance of the H/E ratio in wear control: A nanocomposite coating approach to optimised tribological behaviour, *Wear*, Vol. 246, pp. 1-11, 2000.
- [2] J. Jiang, R.D. Arnell: The effect of substrate surface roughness on the wear of DLC coatings, *Wear*, Vol. 239, pp. 1-9, 2000.
- [3] F. Svahn, Å. Kassman-Rudolphi, E. Wallén: The influence of surface roughness on friction and wear of machine element coatings, *Wear*, Vol. 254, pp. 1092-1098, 2003.
- [4] P. Panjan, P. Gselman, D. Kek-Merl, M. Čekada, M. Panjan, G. Dražić, T. Bončina, F. Županič: Growth defect density in PVD hard coatings prepared by different deposition techniques, *Surface and Coatings Technology*, Vol. 237, pp. 349-356, 2013.
- [5] S.J. Bull: Failure mode maps in the thin film scratch adhesion test, *Tribology International*, Vol. 30, pp. 491-498, 1997.
- [6] D. Kakaš, P. Terek, L. Kovačević, A. Miletić, B. Škorić: Influence of interfacial layer thickness and substrate roughness on adhesion of tin coatings deposited at low temperatures by IBAD, *Surface Review and Letters*, Vol. 18, pp. 83-90, 2011.
- [7] P.C. Wo, P.R. Munroe, Z.F. Zhou, K.Y. Li, Z.H. Xie: Effects of TiN sublayers on the response of TiSiN nanocomposite coatings to nanoindentation and scratching contacts, *Material Science and Engineering A*, Vol. 527, pp. 4447-4457, 2010.