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RESEARCH

Influence of Load and Sliding Speed on Friction Coefficient of IBAD Deposited TiN

The influence of applied load and sliding speed on the tribological performance, i.e. friction and wear of TiN IBAD coating in sliding with corundum ball has been evaluated using reciprocating sliding wear test. Post characterization of wear zones was conducted using AFM, SEM and EDX. The results show that coefficient of friction decrease with decreasing applied load and with increasing the sliding speeds.

Keywords: *Friction coefficient, TiN, AFM, reciprocating sliding, SEM*

1. INTRODUCTION

Tribology phenomena are very dependent on applied load, mechanical properties of contact materials, surface topographies and are different at macro- and nanoscale [1]. Nanoscale friction and wear appear rarely in everyday industry conditions. Although high precision machining has been constantly developed implying smaller contact loads, observed tribology phenomena are not typical for nanotribology.

Wear which appears during high and ultra precision machining is in the scale of several hundred nanometers. In order to observe such a small wear, friction should be decreased and surface properties, such as nanohardness should be improved [2, 3].

The goal of this research was to investigate influence of applied load and sliding speed on the friction and wear during reciprocating sliding of TiN coating against corundum ball. The range of applied loads was carefully chosen in such a way to induce wear depths not larger than several hundred nanometers. There are different types of devices used for evaluation of tribological properties. In this research the CSM Nanotribometer was chosen, while it offers loads in milli-Newton range and broad range of sliding speeds [4, 5].

The TiN coatings were prepared by ion beam assisted deposition (IBAD). IBAD refers to the process in which films produced by physical vapor deposition (PVD) are simultaneously bombarded by the independently generated flux of ions. The extra energy imparted to the deposited atoms enhances their migration along the surface which results in smooth as-deposited surfaces [6].

An atomic force microscope (AFM) was used to observe a surface morphology of as-deposited TiN coating and worn zones generated during tribotests. In order to better understand friction and wear it is recommended to implement scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX) in the study [7, 8].

2. MATERIALS AND EXPERIMENT

TiN coatings were deposited in an Ion Beam Assisted Deposition (IBAD) chamber with a base pressure of 1.5×10^{-6} mbar. The used apparatus consists of a 5 cm Kaufman ion source, 5 kW e-beam evaporator, residual gas analyzer, thickness monitor, and mechanical and cryo pumps. Carburizing steel (0.165%C, 0.2%Si, 1.2%Mn and 1%Cr) disks were used as substrate material. Cemented steel was used due to its high load bearing capacity. Surface roughness of the samples after deposition was measured by AFM and it has values around 4nm of average roughness, Figure 1. Such smoothness that is achieved is a consequence of proper selection of ion incident energy and atom/ion ratio, what is a few years experience of research in National Laboratory Nikola Tesla Vinča. Friction and wear behavior was evaluated by

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ball-on-block tests in dry sliding conditions. The tests were conducted at CSM Nanotribometer.

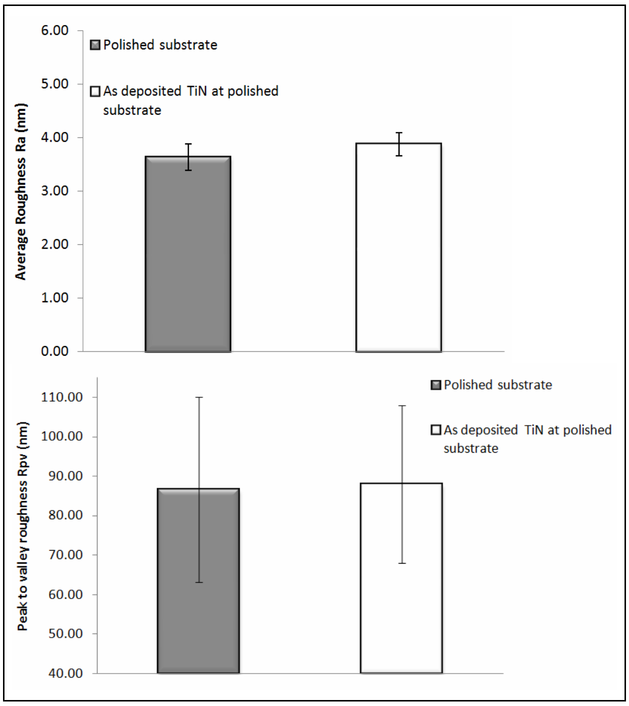


Figure 1. Surface roughness of the samples before and after deposition

The following parameters were used: counter material – 2 mm Al_2O_3 ball with hardness of 2700 HV, movement type – linear reciprocating movement, half amplitude 0.5 mm, total sliding distance 3000 mm, normal load 100 mN and sliding speed 10 mm/s.

Surface roughness and morphology were evaluated by VEECO di CP II atomic force microscope. All images were required in contact mode by etched silicon probe symmetric typ. The size of scanned surface area was 90 x 90 µm. Scanning electron microscopy (SEM JEOL JSM 6460 LV with an embedded Oxford Instrument EDX analyzer) was applied to investigate worn zone morphology.

3. RESULTS AND DISCUSSION

Table 1 presents the average values of the friction coefficient of the TiN coating sliding against corundum ball at three different normal loads and three different sliding speeds.

Table 1. Average friction coefficients of TiN sliding against Al_2O_3 in the ball-on-plate tests

Friction coefficients	$F_1 = 100$ mN	$F_2 = 500$ mN	$F_3 = 1000$ mN
$v_1 = 10$ mm/s	0.169	0.180	0.185
$v_2 = 15$ mm/s	0.160	0.168	0.178
$v_3 = 25$ mm/s	0.134	0.165	0.176

It is obvious that increase in normal load resulted with slight increase of friction coefficient, Figure 2. Influence of sliding speed is also visible but less obvious than influence of normal load.

During sliding of the ball on TiN surface, with low speeds, there is present a lot of oscillations so minimum and maximum values are very different compared to mean value, Figure 3. Mean value of friction coefficient starting from 0,125 and after very short period is increased to a value of 0,169. Near the same value of friction coefficient could be find if it was applied ten times higher load and 2,5 times higher sliding speed (see figure 2.). It could be explained with influence of stick-slip effects at tribo-pair contact surface. Measuring by AFM prove that starting quality of surface is excellent respecting extremely low value of R_a due to the polishing before TiN deposition.

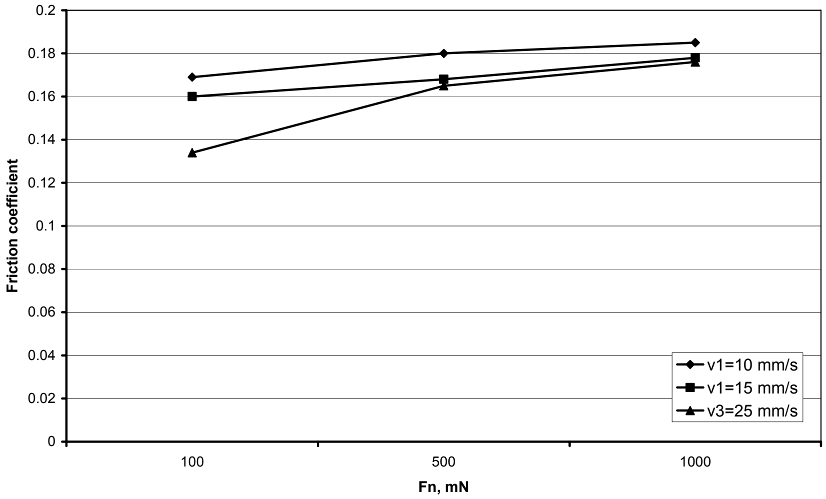


Figure 2. Influence of sliding speed and normal load on friction coefficient

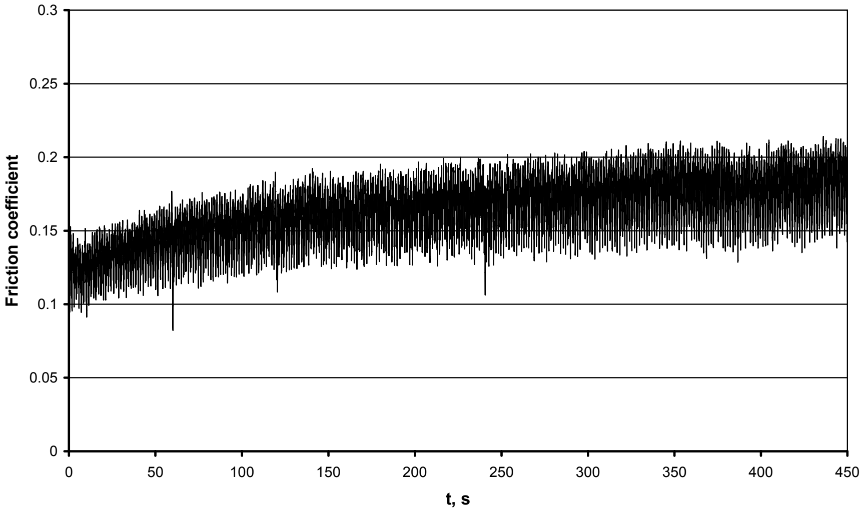


Figure 3. Evolution of friction coefficient during sliding, load F=100mN, speed v=10 mm/s

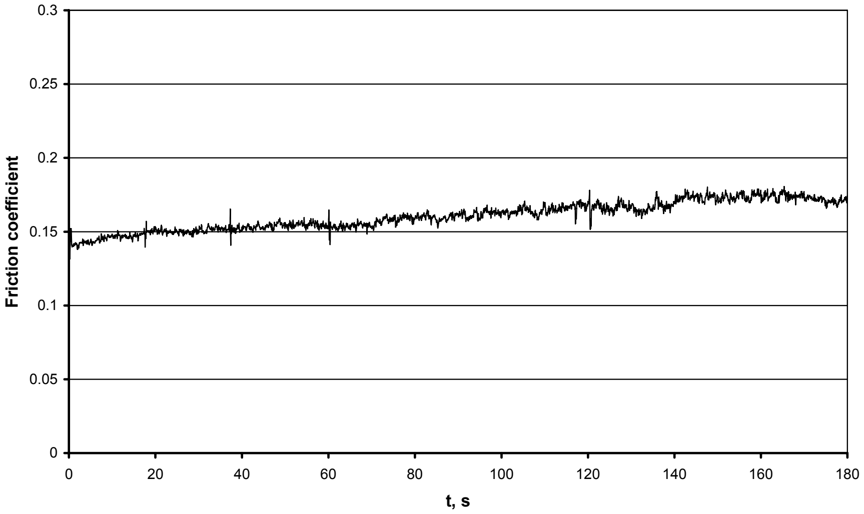


Figure 4. Evolution of friction coefficient during sliding, load F=1000 mN, speed v=25 mm/s

The results of the SEM analysis of worn surfaces for different applied load are presented in Figure 5 and Figure 6. It is clearly visible that the width of the worn channel is different. Result of EDX analyses are presented in Table 2. and Table 3. Applied ion energy of 10keV resulted with penetration into TiN layer till 800-1000nm. Beside titanium and nitrogen, some amount of oxygen was present. The higher oxygen content was measured at the bottom of the worn channel in both cases (Table 2 and Table 3). The higher presence of oxygen is attributed to high contact temperatures which promote surface oxidation [9,10]. An increase of local temperatures between contact tribological surfaces is result of friction taking place at very small contacts.

Table 2. Results of EDX analysis, Figure 5

	Ti	N	O
Spectrum 1	79.18	17.13	3.69
Spectrum 2	75.99	16.09	7.92
Spectrum 3	75.80	14.75	9.45

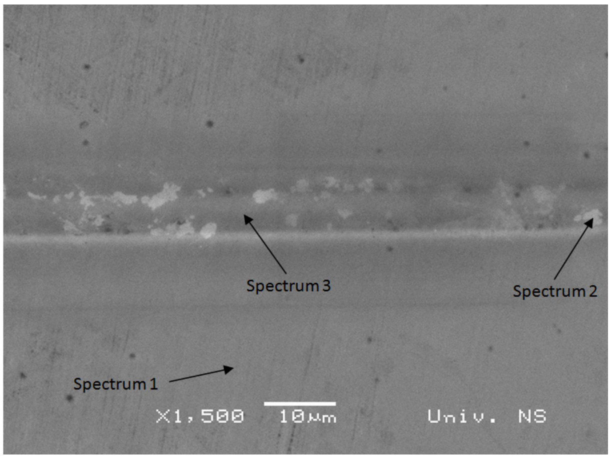


Figure 5. SEM analyze of worn surface, load F=100 mN, speed v=10 mm/s

Respecting that counter body is aluminum oxide ball it could be expected that EDX analysis has to show some presence of aluminum in the worn channel assumed that wear process produce some debris. The applied energy of EDX analysis was too high to acquire data from depths smaller than

50 nm. Regarding that any trace of aluminum was not found, it appears that material transfer from corundum ball did not happen, or that the formed debris was very thin to observe their presence.

Table 3. Results of EDX analysis, Figure 6. a)

	Ti	N	O
Spectrum 1	78.34	15.19	6.48
Spectrum 2	75.41	13.88	10.71
Spectrum 3	74.84	14.50	10.65
Spectrum 4	78.26	15.97	5.77

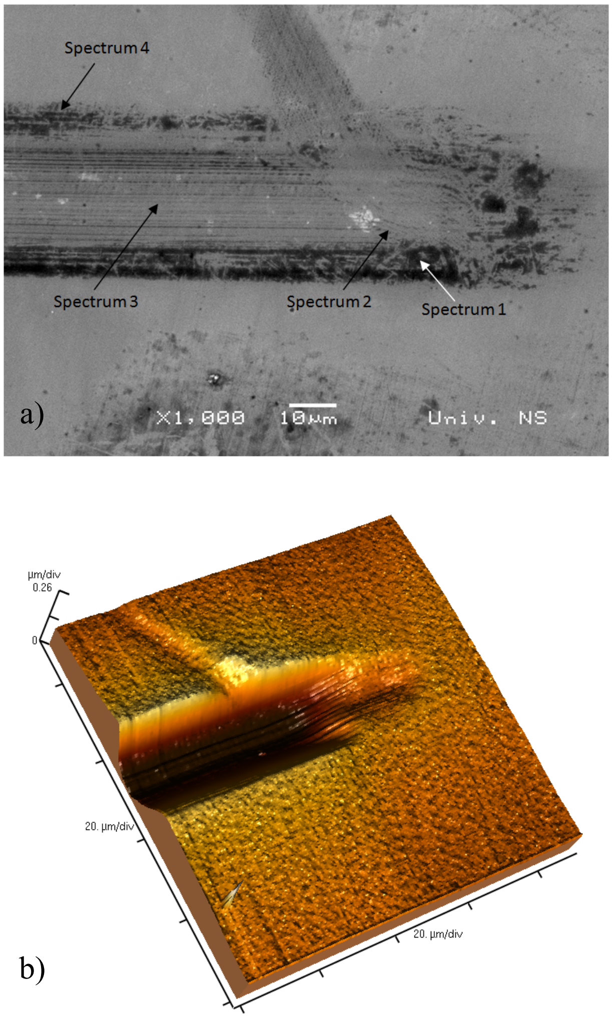


Figure 6. a) SEM analyze of worn surface – Load 1000mN, speed v=10 mm/s, b) accompanying 3D-AFM image

To clarify this it is necessary to use AFM measuring. The 3D images of these two channels are presented at Figure 7 and Figure 8. It is obvious that absence of significant concentration of wear debris inside of channel or near the channel.

Thorough analysis can be made by using of great palette of possibilities of AFM imaging software (Image Processing). It was observed that wear of TiN coating increases with increasing the sliding speed, while the decrease of friction coefficient was observed, which is in consistence with [9,10].

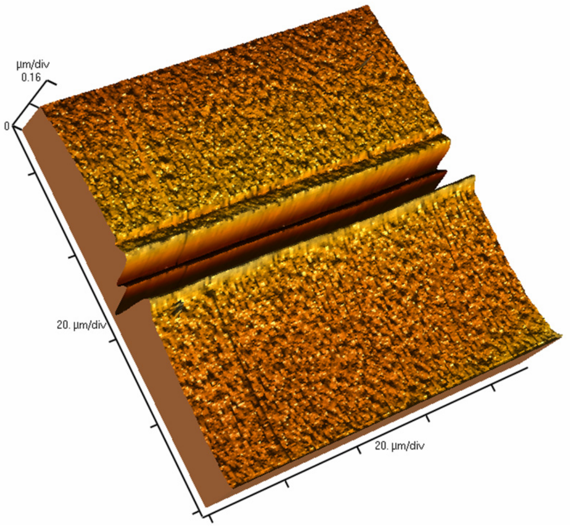


Figure 7. 3D-AFM image of worn surface, load F=100 mN, speed v=10 mm/s

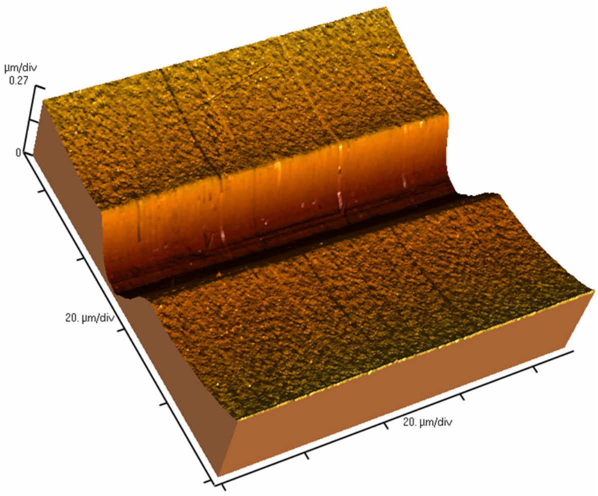


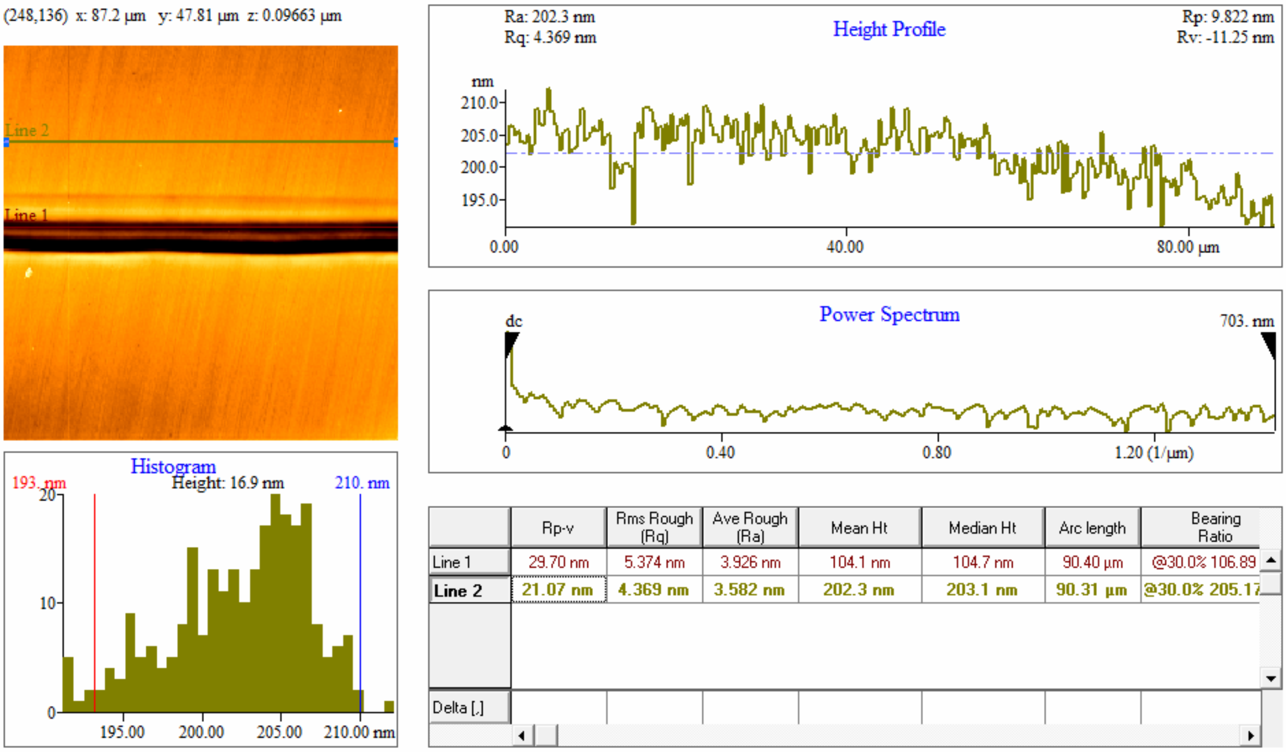
Figure 8. 3D-AFM image of worn surface, Load 1000mN, speed v=10mm/s

In the following, samples subjected to load of F=100 mN and sliding speed of v=10 mm/s will appeal as Zone 1 and samples subjected to load of F=1000 mN and speed v=10 mm/s will appeal as Zone 2. Line analysis at the worn surface (Zone 1) shows very similar roughness of the bottom of the worn channel (Ra=3,926) compared to the starting surface roughness (Ra=3,582), Figure 9.

Figure 10 and 12 show cross-section profile lines of the wear channels, the observed smooth lines are typical for wearing the flat surface against hard ball. The line analysis allows for precise determination of the depth and the width of wear channels, which have values of 223 nm and 10.2 µm respectively for the channel shown in the Figure 10 and Figure 11. The differences of wear tracks produced during different sliding conditions can be observed by comparing Figures 10-13. It is obvious that the depth and the width of wear channels increase with increasing applied load.

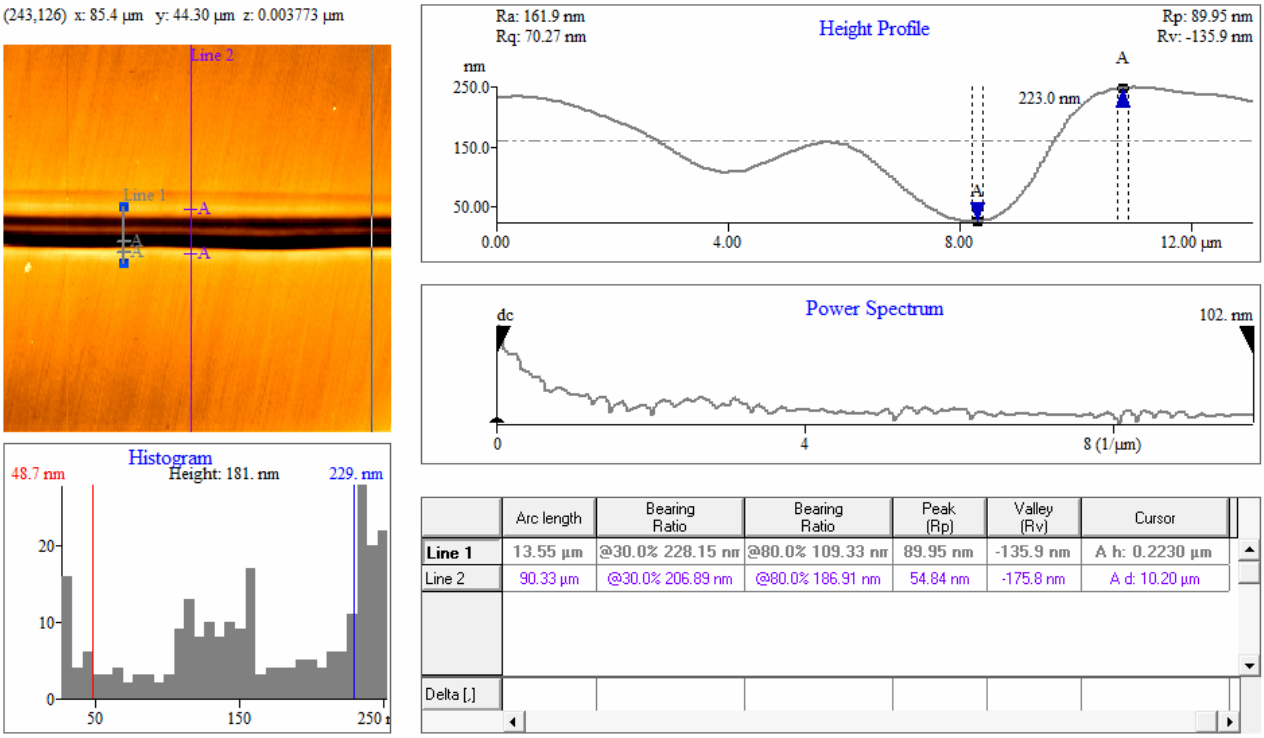
The average roughness at the bottom of the wear channel (R_a=4,174) shown in Figure 12 is close to the surface starting roughness (R_a=3,588).

The presence of wear debris in zone 2 would contribute to increase in peak-to-valley roughness. Considering that, the peak-to-valley roughness was twice smaller at the worn channel comparing to same roughness of as-deposited surface (Figure 12), it was suggested that no significant wear debris were formed at the wear channel.



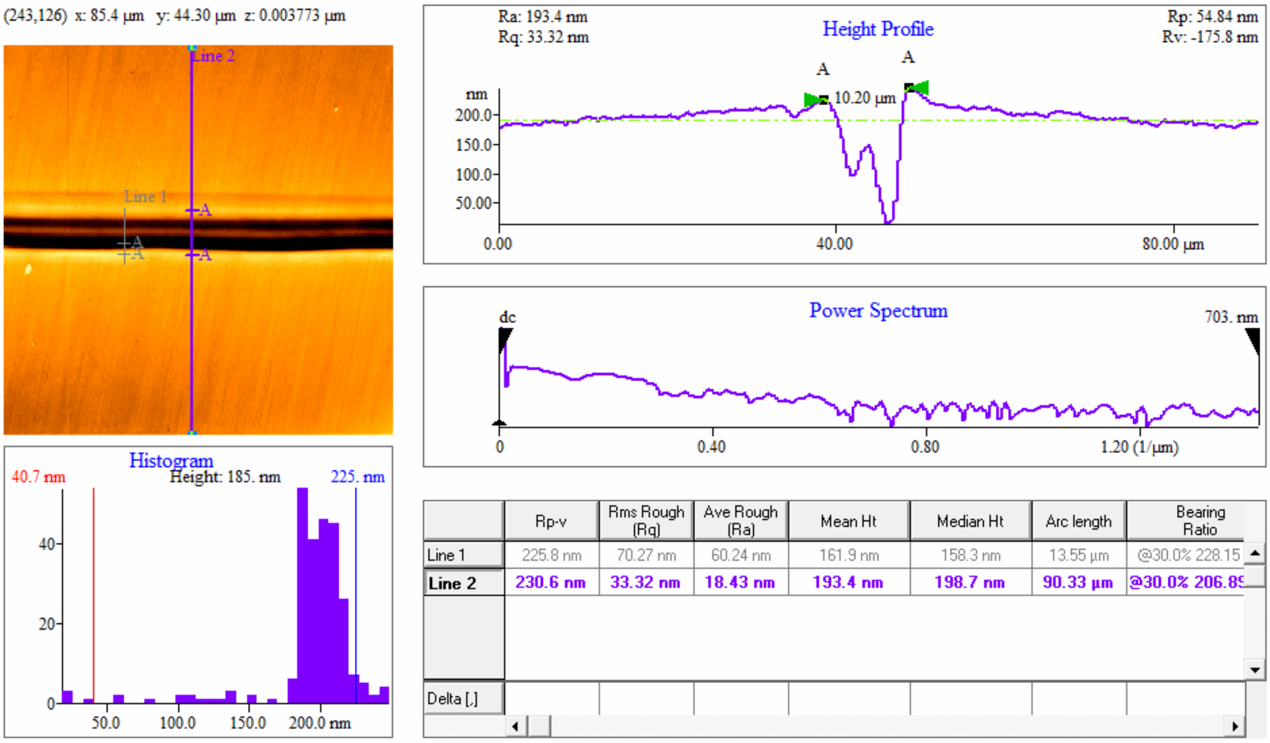
	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Arc length	Bearing Ratio
Line 1	29.70 nm	5.374 nm	3.926 nm	104.1 nm	104.7 nm	90.40 µm	@30.0% 106.89
Line 2	21.07 nm	4.369 nm	3.582 nm	202.3 nm	203.1 nm	90.31 µm	@30.0% 205.17
Delta [,]							

Figure 9. Line analysis along the worn channel (Zone1)



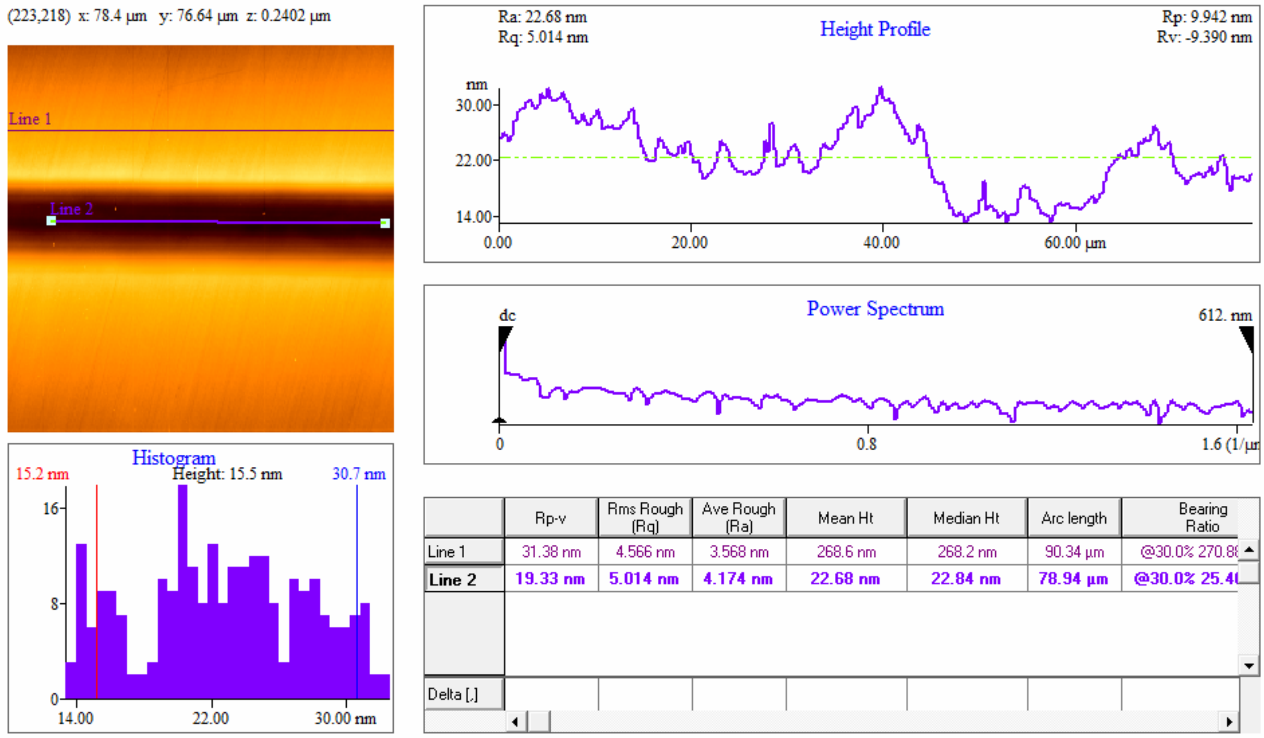
	Arc length	Bearing Ratio	Bearing Ratio	Peak (Rp)	Valley (Rv)	Cursor
Line 1	13.55 µm	@30.0% 228.15 nm	@80.0% 109.33 nm	89.95 nm	-135.9 nm	A h: 0.2230 µm
Line 2	90.33 µm	@30.0% 206.89 nm	@80.0% 186.91 nm	54.84 nm	-175.8 nm	A d: 10.20 µm
Delta [,]						

Figure 10. Line analysis across the worn channel (Zone1)



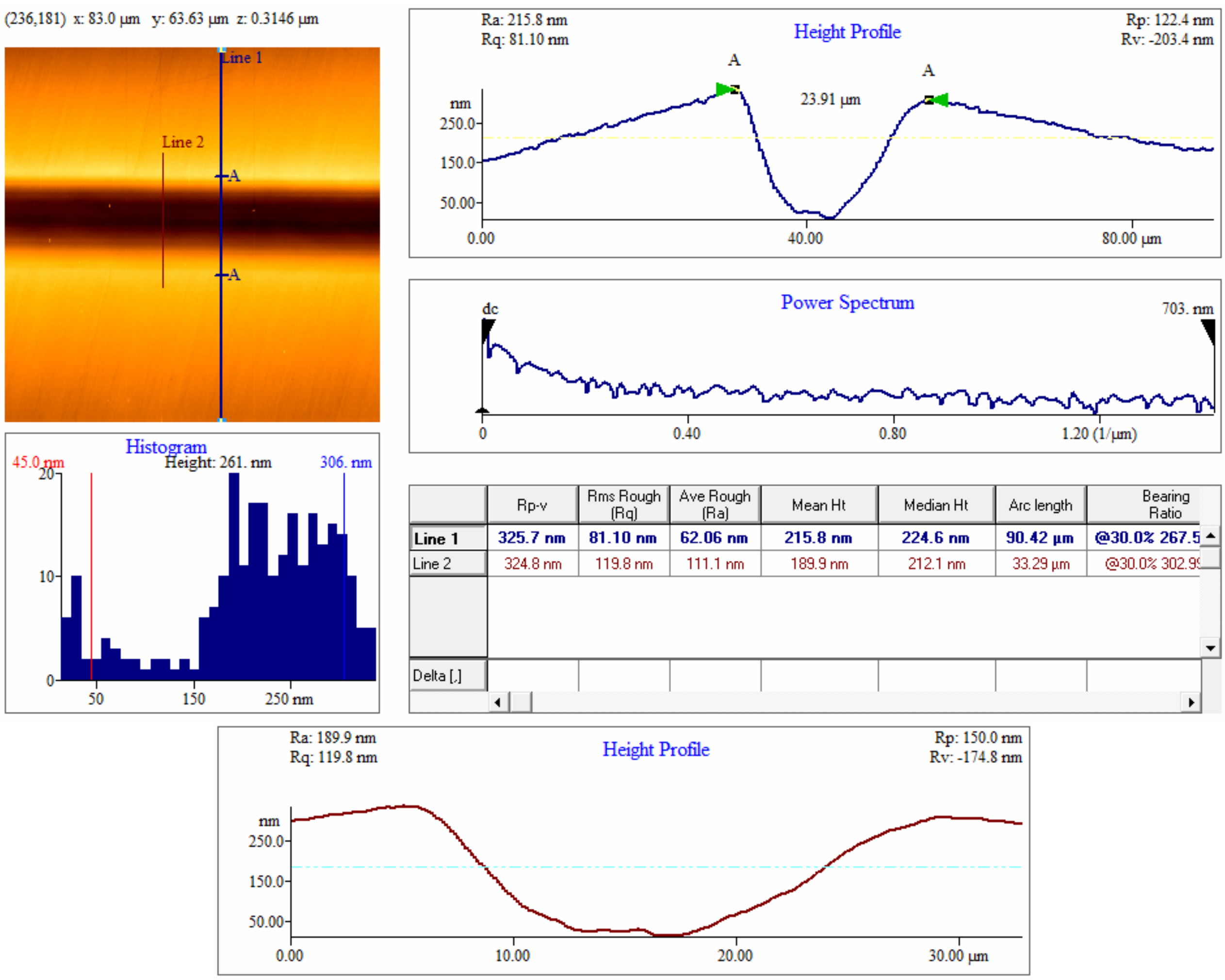
	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Arc length	Bearing Ratio
Line 1	225.8 nm	70.27 nm	60.24 nm	161.9 nm	158.3 nm	13.55 µm	@30.0% 228.15
Line 2	**230.6 nm**	**33.32 nm**	**18.43 nm**	**193.4 nm**	**198.7 nm**	**90.33 µm**	**@30.0% 206.8**
Delta [.]							

Figure 11. Line analysis across the worn channel (Zone1), Line2



	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Arc length	Bearing Ratio
Line 1	31.38 nm	4.566 nm	3.568 nm	268.6 nm	268.2 nm	90.34 µm	@30.0% 270.8
Line 2	**19.33 nm**	**5.014 nm**	**4.174 nm**	**22.68 nm**	**22.84 nm**	**78.94 µm**	**@30.0% 25.4**
Delta [.]							

Figure 12. Line analysis along the worn channel (Zone2)



	Rp-v	Rms Rough (Rq)	Ave Rough (Ra)	Mean Ht	Median Ht	Arc length	Bearing Ratio
Line 1	**325.7 nm**	**81.10 nm**	**62.06 nm**	**215.8 nm**	**224.6 nm**	**90.42 µm**	**@30.0% 267.5**
Line 2	324.8 nm	119.8 nm	111.1 nm	189.9 nm	212.1 nm	33.29 µm	@30.0% 302.9
Delta [,]							

Figure 13. Line analysis across the worn channel (Zone2)

4. CONCLUSIONS

The tribological properties of TiN films prepared by ion beam assisted deposition (IBAD) were evaluated. Although IBAD is technology which influences the roughness of thin films greatly, the difference between the surface roughness of steel substrate and as-deposited TiN coatings was negligible.

Change in applied load and sliding speed influenced the average values of friction coefficient. Low fluctuation of COF was observed when sliding with high speeds.

The EDS analysis confirmed the presence of oxides at the worn surface. There was no evidence of presence of aluminum inside wear tracks. It was supposed that the material transfer from the corundum ball did not happen because the ball was harder than TiN coating.

Atomic force microscopy shows no significant disturbances, nor smeared debris inside the wear track, and gives the explanation for low values of friction coefficient.

The depth and width of wear track increased with increasing applied load, while COF did not changed significantly.

Relatively small difference in the COF at different applied loads can be explained by different oxidation of contact surfaces, which influences sliding properties of contacting surfaces.

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