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SURFACE TOPOGRAPHY INVESTIGATIONS OF TiN LAYERS ON DIFFERENT SUBSTRATES

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Abstract. We have performed the studies of the surface topography of TiN layers deposited on steel and silicon substrates. TiN coatings have been prepared by the arc physical vapor deposition (PVD) technique. As the final useful mechanical properties of protective coatings are strongly determined by the state of the surface, we have carried out the complex optical, atomic force microscopy (AFM), and scanning electron microscopy (SEM) investigations. The optical studies include *XY* optical profilometer measurements and bidirectional reflection distribution function (BRDF) measurements, being complementary to AFM and SEM methods. From the power spectral density (PSD) function obtained from the optical data, the root-mean square (rms) roughness and correlation length have been determined. This has allowed one to estimate correlation between roughness of surface before and after deposition and to measure the surface parameters.

1. INTRODUCTION

TiN is a well known material used in many industrial applications and, in particular, as hard protective coating layers on metallic or dielectric substrates (cutting tools). In this work we have studied the surface topography of TiN layers deposited on steel and Si using the optical methods along with AFM, SEM, and mechanical profilometry investigations.

One of the crucial parameters of protective coatings is the surface roughness. Therefore, various models have been developed for surface roughness assessment characterizing real surfaces [1,2]. In general, the basic information is obtained from an analysis of the surface power spectral density (PSD) function. PSD expresses the roughness power per unit roughness frequency over the sampling length [3] and it is evaluated from the Fourier transform of the surface profile. The PSD function is commonly determined from mechanical profilometry measurements or by processing AFM and SEM images.

Roughness of layers is usually quantified by analyzing the surface-profile data in order to extract various statistical averages in specified spatial bandwidths. The most accurate approach for the surface statistic determination is calculation of PSD function from these profiles is a function of spatial frequency f_x [4]. This yields the required bandwidth and allows one to determine contributions of different spatial wavelength irregularities $S_1(f_x)$ to the total roughness. The statistical averages of roughness σ and slopes m are calculated from PSD frequency spectra by the following formulae

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$$\sigma^2 = 2\int_{f_{\min}}^{f_{\max}} (2\pi f_x)^0 S_1(f_x) df_x, \quad (1)$$

$$m^2 = 2\int_{f_{\min}}^{f_{\max}} (2\pi f_x)^0 S_1(f_x) df_x. \quad (2)$$

The correlation length is simply the ratio

$$T = \frac{\sqrt{2}\sigma}{m}. \quad (3)$$

A powerful tool for determination of topographic parameters of a measured surface is called the Bidirectional Reflectance Distribution Function (BRDF) [4]. BRDF relates the differential power of scattered beam *dP*, per differential solid angle of receiver aperture $d\Omega$ in the θ_S direction and per incident power P_i coming from the θ_i direction. Practically $dP/d\Omega$ is equal to the measured scatter power P_s per acceptance angle Ω of a detector and then

$$BRDF = \frac{dP / d\Omega}{P_i \Omega \cos\theta_s} \left[sr^{-1}\right]. \quad (4)$$

The Raleigh-Rice vector perturbation theory relates the scattered power spectral density per unit incident power to the power spectral density to give

$$BRDF = \frac{16\pi^2}{\lambda^2} \cos\theta_i \cos\theta_s Q S(f_x), \quad (5)$$

where Q is a factor dependent on the polarization state of the light source and λ is the light wavelength.

If measurements are performed in plane of incidence and incident light is *s*-polarized, Q is given by the Fresnel coefficients at θ_i and θ_S of the specular reflectance. Then

$$Q_s = \{R_s(\theta_i) R_s(\theta_s)\}^{\frac{1}{2}}. \quad (6)$$

For highly reflective metallic reflectors, the last formula can be substituted by $Q_s = R_s(\theta_i)$ which is simply the specular reflectance at angle of incidence θ_i. It is also very convenient to determine PSD from Eqs. (6) and (7) without knowing the optical constants of sample. This means that BRDF and PSD (except for the factor $Q\cos\theta_S$) are directly proportional. The formula (5) is a principle of surface investigations by the optical scatterometry and also gives the rms surface roughness (σ_{rms}) for a given wavelength (even for $\sigma_{\mathrm{rrns}} \leq 0.5\lambda$) and adequately large angle of incidence.

In this work, we have studied TiN coatings deposited on high speed steel and Si using the optical methods described above. For completeness, also AFM and SEM methods have been used. This has allowed one to examine the surface topography of samples and to determine the surface parameters at different spatial wavelength (different spatial frequencies). Taking the surface morphology of substrates into consideration, we have studied the TiN coating roughness and their correlation with ground materials.

2. EXPERIMENTAL

We have studied the TiN coatings deposited on mechanically polished SWM7 high speed steel substrates with the same polishing time and Si substrates with different roughness. The TiN layers have been obtained using the physical vapor deposition (PVD) method. Prior to the covering, the tools have been cleaned by glow discharge in nitrogen atmosphere. After cleaning, a short-time titanium bombardment have been applied. The bias voltage has been of 1 kV. The whole procedure has been performed in a commercial equipment PUSK 83 [5]. The final coating with TiN has been obtained by arc titanium cathode discharge in nitrogen atmosphere. The preliminary treatment with the above-mentioned equipment plays an important role in durability of tools covered with TiN [6]. Also the surface roughness of the substrate influences quality of the TiN coatings [7].

BRDF measurements were performed with an automatic home-made scatterometer. It consists of a 650 nm laser diode as a light source with the beam diameter of 2 mm mounted on a goniometric table with 0.1 deg resolution. The light scattered at the sample surface is measured with a Si photodiode detector. The rotations are obtained by a computer controlled stepper motors. For a fixed angle of incidence, the scattered intensity in the plane of incidence was measured by varying the detector orientation. All measurements have been carried out with the *s*-polarized incident beam. In any case, the sample surface size was much larger than the beam diameter. Moreover, the minimal illuminated area (4 mm^2) has been large enough to yield meaningful statistical description of the surface.

Measurements of optical reflectance by means of the classical reflectometry inform us about optical properties on a large area, i.e. of the order of 1-5 cm^2. The results obtained on a much less scale will be similar if coatings and surfaces are homog-

Table 1. Roughness of the samples studied.

Sample	Sample description	Microroughness σ_{ss} (nm)	Total roughness σ_{tot} (nm)
S1	Bare SW7M	279	3700
S2	TiN on SW7M (30 min)	310	310
K1	polished Si	1	2
K2	rough Si	736	800
K3	TiN on polished Si (2 min)	110	100
K4	TiN on polished Si (20 min)	404	400
K5	TiN on rough Si (20 min)	390	400

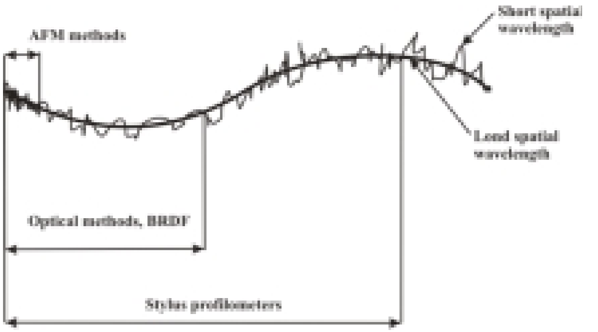

Fig. 1. Scheme of surface profile with short and long spatial wavelengths.

enous over the investigated area and inside the layers. For inhomogeneous surfaces, when topographic or materials nonuniformities differ from tens μm to several mm, the measurements taken from the sphere and from reflectometer give rather an averaged reflectance over a larger scale reflected samples. In goal to get reflectance over smaller areas, the reflection probe R200-7 has been used (see e.g. [8]). The probe was mounted on *XY* positioning stage. A commercial lead screw stepper motor actuated device has been used for scanning 10 mm x 10 mm surface with steep of 0.1 mm. Optical profilometry (OP) measurements have been normalized with the calibration sphere method [9]. It allows one to obtain the optical map of topography with 0.1 mm lateral resolution. These measurements complete the morphology description between micro (AFM and SEM) and macro (BRDF) spatial wavelengths.

3. RESULTS AND DISCUSSION

Roughness of the samples studied, including substrates (S1, K1, and K2), is presented in Table 1. Column 2 of the table contains (in brackets) also time of TiN deposition (S2, K3, K4, and K5). The short spatial microroughness σ_{ss}, shown in column 3, has been measured in a small spot area, i.e. with AFM and SEM. In column 4, the values of total roughness σ_{tot}, determined on a much larger surface area by BRDF and optical or stylus prolilometry, have been shown. The total roughness can be expressed as $\sigma_{tot} = \sqrt{\sigma_{ss}^2 + \sigma_{ls}^2}$, where σ_{ls} denotes long spatial roughness originating from periodic mechanical polishing as shown in Fig. 1.

The SWM7 substrate (sample S1) was mechanically polished prior to the TiN deposition. This kind of polishing gives a periodic surface with grooves. For such surfaces the BRDF measurements performed on a large scan area yield much greater roughness than that detected in small area AFM scans.

Fig. 2 shows the angular dependence of BRDF for samples S1 and S2. The peak visible on curve S1 is a result of light scattering on the surface grooves. The spacing *L* of the grooves can be derived from the peak positions occurring at angles given by

$$\sin(\theta_{scat}) = \sin(\theta_{inc}) + \frac{\lambda}{L} \qquad (7)$$

and yielding L = 189 μm. The height of peaks indicates how dense and deep grooves are; larger peaks indicate deeper surface grooves. After adequately long time of TiN deposition (sample S2), the peak originating from grooves practically disappears (Fig. 2, curve S2). Thus, it could be concluded that TiN fills the grooves and levels out the

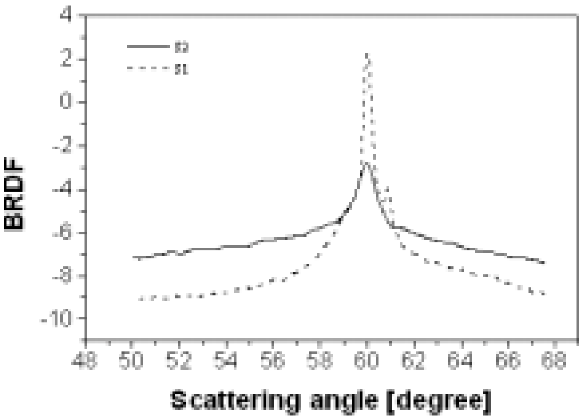

Fig. 2. BRDF vs. scattering angle θ_S for samples S1 and S2.

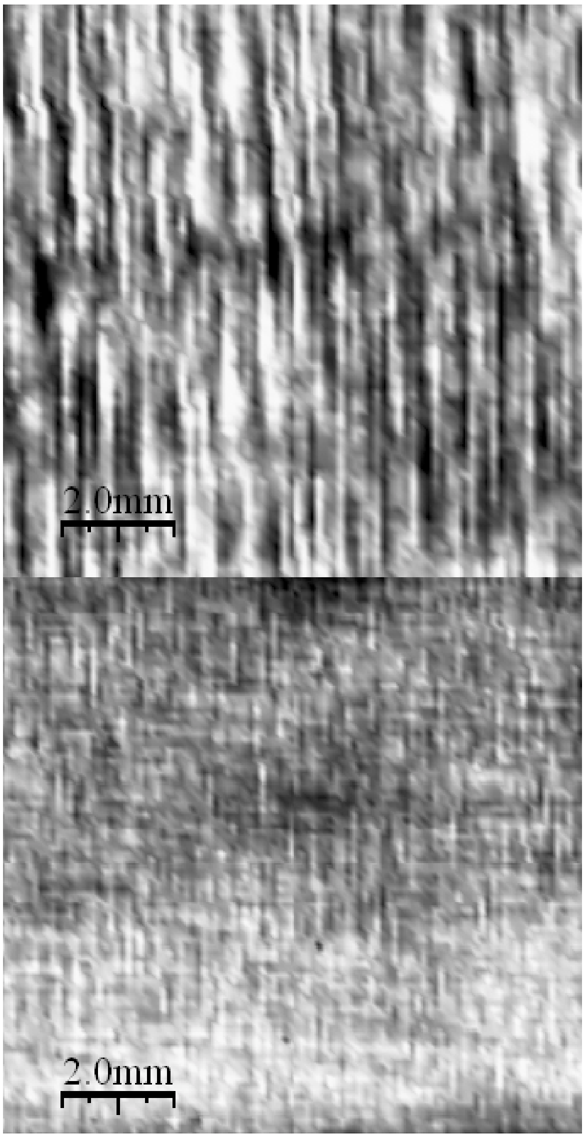

Fig. 3. Surface topography for samples S1 and S2 obtained with the optical profilometry.

surface. Time of TiN deposition longer than 30 min does not change essentially the roughness.

It is evident, that the N_2 glow discharge cleaning process and subsequent Ti bombardment alter significantly the surface roughness of most samples. However, the roughness changes depend on the surface topography of substrates (their initial surfaces and slopes) and, what is less obvious, on nature of these surfaces (random or periodic).

Figs. 3a and 3b show the scanned surface profiles of samples S1 and S2 obtained from optical profilometry (OP) and corresponding to the appropriate curves in Fig. 2. The grooves with the spatial length determined from BRDF are also visible for sample S1 in Fig. 3a and they disappear for sample S2 (Fig. 3b) evidently confirming the BRDF results. Additionally, the OP data have allowed us to calculate the root-mean square roughness σ_{rms} in the long spatial wavelength (0.1-10 mm) range. It gives a very large contribution to the total roughness, namely 3.6 μm for sample S1.

The AFM images of fine polished Si substrate (sample K1) and the same substrate covered with TiN layer (samples K3 and K4) are presented in Fig. 4. Values of roughness obtained from BRDF (column 4 of Table 1) are larger than those obtained from AFM scanning, because the BRDF measurements comprise a wider range of spatial wavelengths. This seems to be reasonable from another point of view: Namely, the AFM image is limited to small (25 μm x 25 μm) local areas, while BRDF data are gathered from the 4 mm^2 illuminated spot. Therefore, optical measurements give information about more global surface irregularities, and roughness results are almost always slightly bigger than AFM ones. But if a surface reveals waviness (in the millimeter range), the results revealed by both methods could differ much more.

Comparing a fine smooth surface substrate (sample K1) with TiN layer deposited on it (sample K3), one can see that the roughness of the latter is

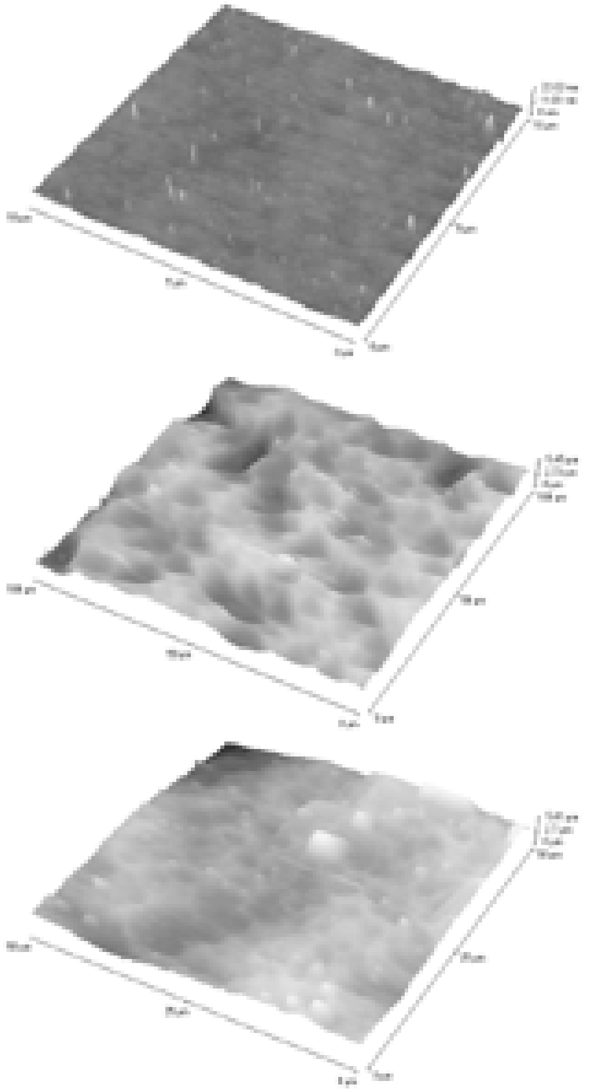

Fig. 4. AFM images for smooth and rough Si substrates and with TiN coatings. Pictures a, b, c correspond to samples K1, K2, and K4, respectively.

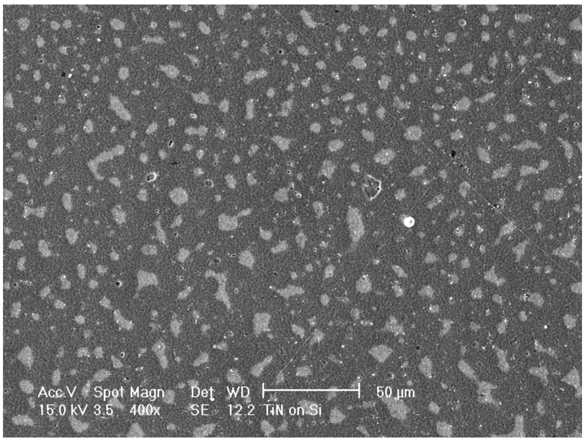

Fig. 5. SEM image of TiN layer on the smooth Si substrate (sample K3).

much bigger than that of the bare substrate. For rough Si substrate (sample K2) with roughness of 0.8 µm, an appropriate long time of TiN deposition (sample K5) results in substantial decreasing of roughness, as shown, apart from Table 1, also in Fig. 4c. Roughness for sufficiently long time of deposition (20 min) gives nearly the same values, independently of the substrate roughness (Table 1, samples K1-K5). On the other hand, the calculated correlation lengths for TiN on smooth and rough Si substrate, are equal to 4.5 and 7 mm, respectively. It could be stated that the surface topography of substrate plays a meaningful role even for thicker TiN layers.

The final roughness values of TiN on Si are bigger than those for coatings on SW7M steel samples. An interesting SEM result for sample K3 with a very short time of deposition is shown in Fig. 5. The visible bright speckles are connected with Si local areas uncovered by TiN. It could be due to the surface topography of substrate (sample K1) which is very smooth and the adhesion of TiN to the substrate, in the beginning, is very small resembling the island-forming process.

4. CONCLUSIONS

Optical measurements of BRDF based on the analysis of scattered radiation and OP have been completed with AFM and SEM studies of the surface morphology of TiN coatings on larger areas. BRDF and OP methods detect long spatial wavelength irregularities which contribute substantially to the total roughness. Values of roughness obtained by BRDF are slightly bigger than those estimated from AFM, in accordance with the applied model. For rougher substrate surfaces, the TiN coatings compensate long spatial irregularities, making the surface smoother. The final values of roughness of TiN layers with long deposition time are independent of the substrate roughness.

Values of roughness of thick TiN coatings on Si substrates are bigger than those of SW7M steel ones. It is also observed that for smoother Si substrate, the final roughness of TiN coating is larger.

It is probably connected with the nucleation process playing an important role at the beginning of deposition.

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