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# Study of TiC/a-C(:H) Coatings Before and After Friction by Nanoindentation

**Elena V. Torskaya, Tamara I. Muravyeva and Juan Carlos Sánchez-López**

**Abstract** This study is focused on the analysis of the surface geometry and elastic properties of carbon-based coatings, which are deposited using a high power impulse (HiPIMS) (pulsed) and direct current (DC) (non-pulsed) magnetron sputtering. The work includes friction tests, microscopic (SEM and AFM) study of the samples surfaces, their roughness before and after friction, indentation of initial and worn surfaces, and estimation of Young's modulus of coatings before and after friction using analytical–numerical modeling. The analysis of nano-roughness by AFM shows that worn tracks are very smooth even for an initially rough surface. DC coatings have similar elastic properties before and after friction. HiPIMS coatings become more compliant after friction, which is probably due to the formation of a thin compliant film at the surface of the coatings.

**Keywords** Coatings · Friction contact · Nanoindentation

## 1 Introduction

Nanoindentation is one of the main methods to study mechanical properties of surface layers especially for the case of coatings, because usually the materials do not exist as bulk ones. Standard indentation by Berkovich pyramid is used to find elastic properties and hardness from the model [1, 2] developed for homogeneous materials. To use this procedure, we should have surfaces with low roughness, and for the case of relatively rigid coatings, the compliance of substrate cannot be ignored. Elastic indentation by smooth sphere can be used for the case of rough

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surfaces of coatings [3]. For worn surfaces, it can be used to study the changes in the elastic properties of thin surface layers after friction [4].

In this study, we analyze surface geometry and elastic properties of carbon-based coatings deposited using high power impulse (HiPIMS) (pulsed) and direct current (DC) (non-pulsed) magnetron sputtering. The work includes friction tests, microscopic (SEM and AFM) study of the samples surfaces, their roughness before and after friction, indentation of initial and worn surfaces, and analysis of Young's modulus of coatings before and after friction.

## 2 Coating Samples and Friction Tests

TiC/a-C nanocomposite coatings (DC1 and DC2) were prepared by magnetron sputtering on AISI M2 polished steel substrates. The TiC/a-C:H nanocomposite coatings (HiPIMS-1 and 2) were deposited on steel DIN 1.2083 disks in an industrial-scale high vacuum deposition chamber fabricated by Thin Film Srl [5]. More details about coating deposition and their chemical composition are given in "Fracture of TiC/a-C(:H) Coatings in Friction Contact" published also in the current issue, and one can also refer to [6, 7].

For the friction tests, we used UMT-3 (CETR) tribotester in "ball-on-disk" contact mode with reciprocating motion using 100Cr6 balls (6-mm in diameter) as counterface. Load of 5 N is applied at a frequency of 5 Hz and track length of 10 mm. The duration of the tribological tests was 1 h. The results of the friction measurements and the width of wear tracks are summarized in Table 1.

## 3 Study of Surfaces Before and After Friction

Here we present the results of the microscopic study of the surfaces before and after friction tests using SEM and AFM. Figure 1 illustrates the differences in the initial surfaces of DC coatings. Both films develop a similar granular structure although smoothed in the DC2 sample.

Wear tracks after friction are smooth and look almost similar for both DC samples. Nano-roughness of the surfaces was evaluated by AFM measurements. Almost all width of wear track is presented in Fig. 2 as AFM measured profile. For

**Table 1** Friction coefficients and width of wear tracks

| Sample | Friction coefficient | Width of wear tracks, mm |
|---|---|---|
| DC1 | 0.20 | 0.11 |
| DC2 | 0.19 | 0.14 |
| HiPIMS-1 | 0.08 | 0.16 |
| HiPIMS-2 | 0.11 | 0.24 |

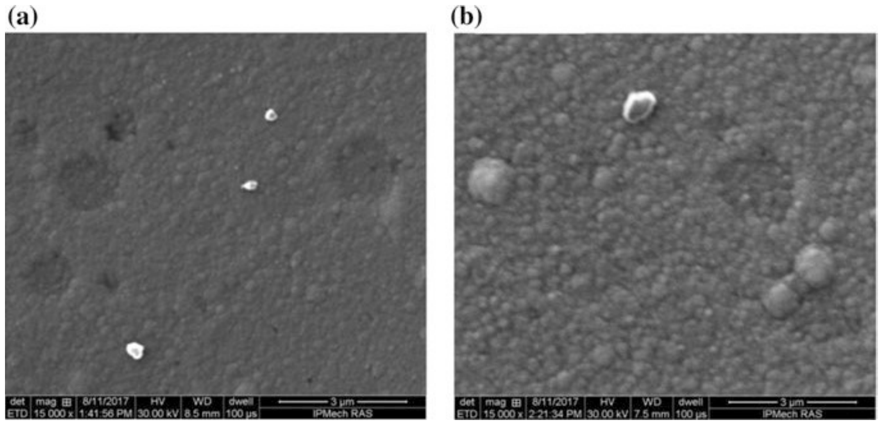


**Fig. 1** SEM micrographs of initial surface of DC1 (**a**) and DC2 (**b**) samples at magnification ×1500

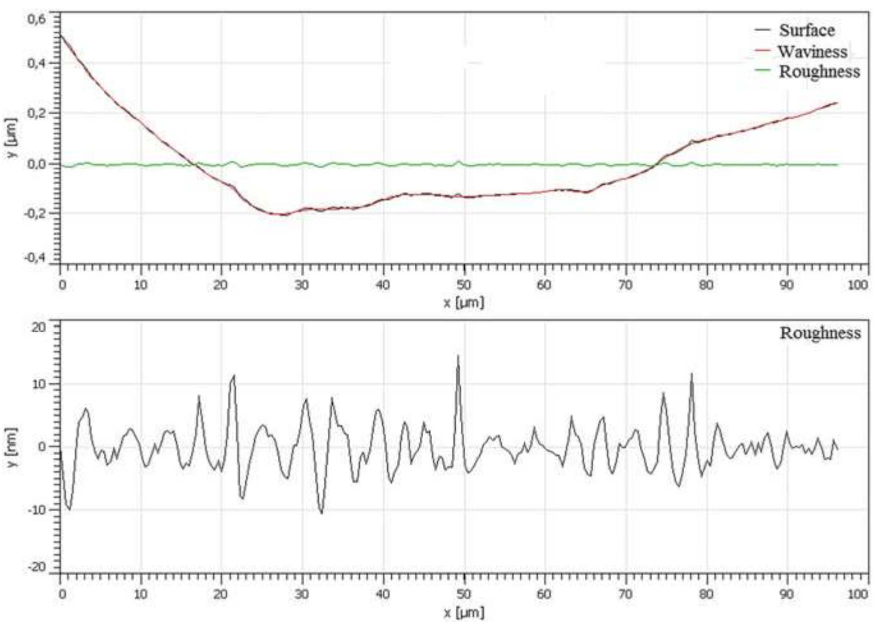


**Fig. 2** AFM profile measurements of DC2 wear track

the case of DC samples, we can resume that roughness is negligible for the worn surfaces as well as for DC2 initial surface, but it should influence the indentation results for DC1 surface before friction. For illustration, we present here surface geometry of DC1 with friction track, which is obtained using profilometry (see Fig. 3).

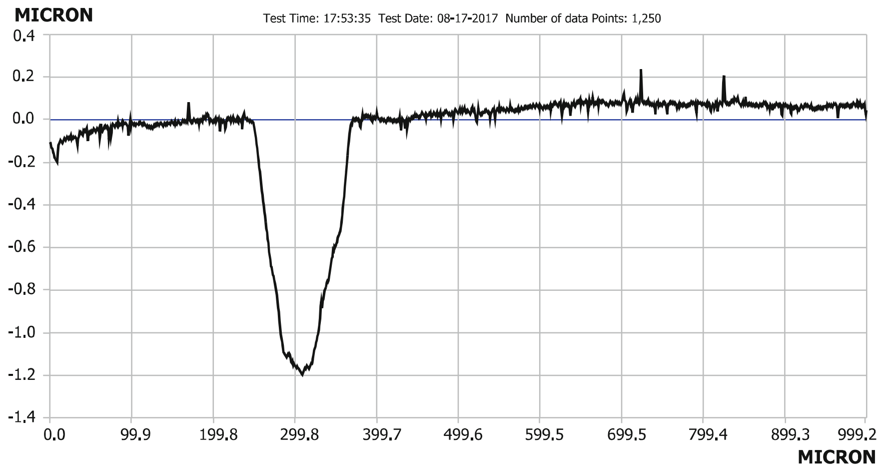


**Fig. 3** Profile of worn surface (DC1)

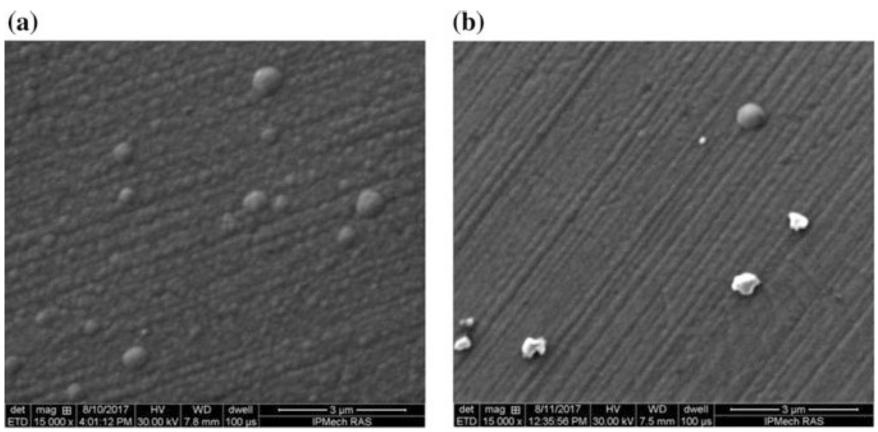


**Fig. 4** SEM micrographs of initial surface of HiPIMS-1 (**a**) and HiPIMS-2 (**b**) samples at magnification $\times 1500$

For the case of HiPIMS coatings, initial surfaces are formed by small grains oriented in regular strips due to the substrate topography, not completely removed during polishing (see Fig. 4). Rounded features are less prominent in the HiPIMS-2 sample. For the contact problem solution, we can consider smooth surface here. After friction, the wear tracks have some local cracks; the track surface without cracks for both HiPIMS coatings also has strips oriented in sliding direction. The roughness of non-cracked worn areas was studied using AFM. The results shown in Fig. 5 demonstrate that the roughness is greater than for DC coatings, but also negligible.

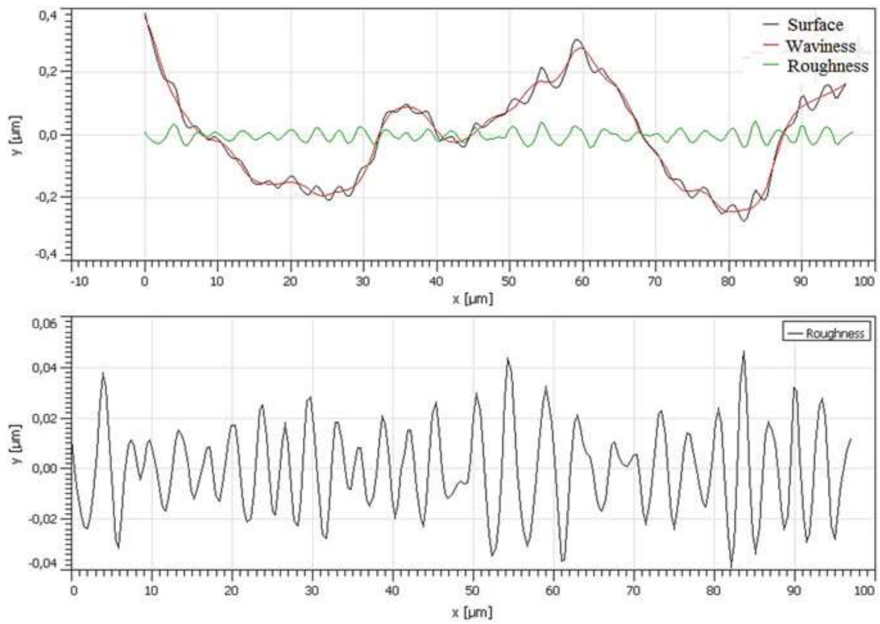


**Fig. 5** AFM profile measurements of HiPIMS-2 wear track

## 4 Indentation Results and Discussion

This section describes the results of testing of the coatings by indentation. In the experiments, we used an indenter of a spherical shape with a diameter of 1.5 mm (alumina ball). Mechanical indentations were done inside and outside of wear tracks formed after friction tests. The width of the friction track was not less than 100 μm (see Table 1). A series of indents was performed with a pitch of 30 μm at an angle of 5° to the direction of the friction track. Thus, for each of the samples, we had 30–40 indents for the tracks. For the experiments, we used Nanoscan 4D [8], which provides a continuous recording of the value of the penetration depth of the indenter and the applied load.

Figure 6 shows the results of a series of tests obtained for HiPIMS-1: (a) initial and (b) worn surfaces. The red lines indicate the averaged curves. Loading and unloading curves are close to each other; it means that we have almost pure elastic indentation, but for the case of tracks, the indentation depth is greater. Such an effect may be the result of different reasons. A first explanation could be an increased roughness during friction, but as it is shown previously, the track is fairly smooth. A decrease in coating thickness due to wear processes would increase the influence of the substrate but steel is less compliant than the coating (200 GPa). The last option is that we have some changes in the elastic properties of the top surface induced by friction.

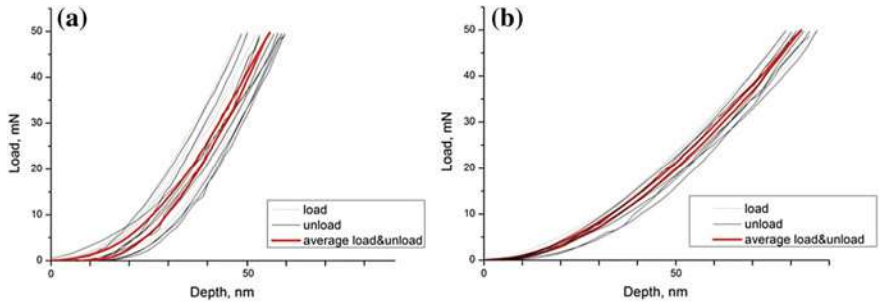


**Fig. 6** Indentation curves for HiPIMS-1 sample: initial surface (**a**) and wear track (**b**)

Indentation results were used for determination of elastic properties of coatings. The coatings are more compliant than the substrate, and the indentation depth is less than 100 nm; it means that we can ignore the substrate deformation during the indentation. We assume that initially, coating materials are homogeneous, and for smooth initial surfaces of DC2 and both HiPIMS samples, we can use classical Hertz contact problem solution to find the elastic modulus of the coatings. We assume that Young's modulus of alumina ball is 600 GPa. For the case of rough surface of the sample (DC1), we can calculate additional compliance due to roughness and use two-level contact model [9]. Contact of the two-layered coating should be considered to check the hypothesis of formation of specific surface layer in friction process, because such a layer should be relatively thin [10]. We use only the results of unloading process with zero load–zero displacement position, which is necessary for modeling.

Figure 7 presents theoretical and experimental results for HiPIMS coatings. Simulated curves of the initial films were obtained with elastic moduli of 82 and 80 GPa for the samples HiPIMS-1 and HiPIMS-2, respectively. Simulation of the curves measured inside the wear track was done for a bilayer structure with a more compliant thin surface layer. We found a good correlation between experimental

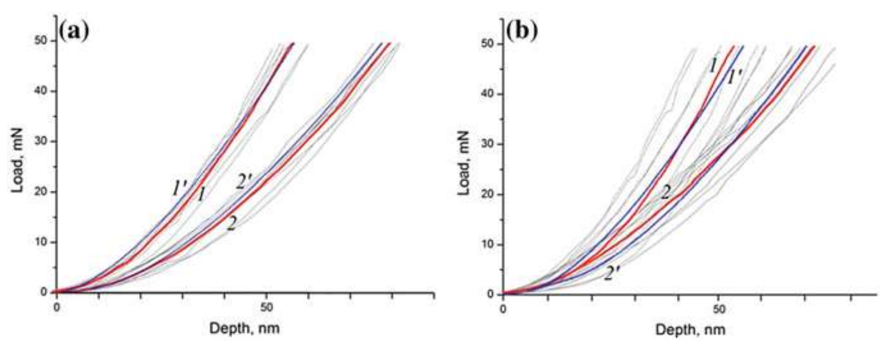


**Fig. 7** Indentation (red curves 1, 2) and computation (blue curves 1′, 2′) for HiPIMS-1 (**a**) and HiPIMS-2 (**b**) samples: initial surface (curves 1, 1′) and friction track (curves 2, 2′)

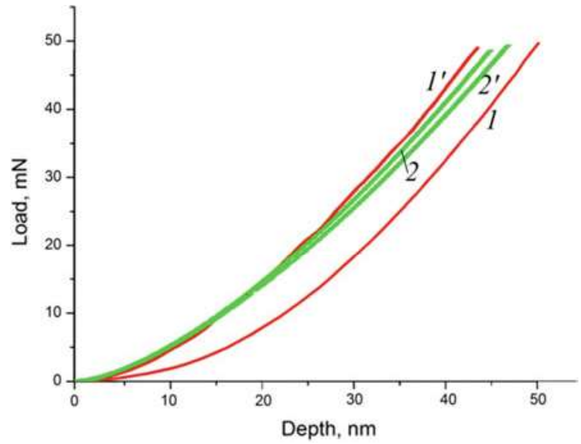


**Fig. 8** Unloading curves for DC2 (1 and 1′) and DC12 (2 and 2′); surface before friction (curves 1, 2) and worn track (curves 1′, 2′)

and theoretical results for a surface layer of 178 nm with an elastic modulus of 26 GPa (HiPIMS-1) and 216 nm with 36 GPa of elastic modulus (HiPIMS-2). We should notice that the results for HiPIMS-2 are less reliable than for HiPIMS-1 due to a larger scattering of experimental data.

Experimental results of indentation tests performed on initial and worn surfaces of DC1 and DC2 coatings are displayed in Fig. 8. DC1 coating surface is smooth initially and also has smooth worn track. The curves 2 and 2′ are very close to each other meaning that there is no friction-induced surface modification in this case. The computation of elasticity modulus resulted in 109 GPa. For the case of DC2 coating, we have a larger penetration depth for the initial surface because of its roughness. Roughness is essential for early stages of indentation (small load); for large loads, the curves have the same angle of inclination. The results correspond to the elasticity modulus of 112 GPa.

## 5 Conclusions

Roughness and elastic properties of two types of carbon-based coatings are studied using the methods of microscopy, nanoindentation, and modeling.

- Analysis of nano-roughness by AFM shows that worn tracks are very smooth even for the case of an initial rough coating (DC2). It can be treated as a sign of gentle friction both for DC and HiPIMS coatings.
- Analytical–numerical methods together with nanoindentation are used to determine elastic properties of surface layer. DC coatings have similar properties before and after friction. For HiPIMS coatings, the phenomenon of formation of thin compliant film at the surface can explain the results of nanoindentation. This phenomenon could be invoked for explaining low and steady-state friction behavior observed with HiPIMS films.

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