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SUBSTRATE INFLUENCE ON THE MECHANICAL PROPERTIES OF TiC/a-C COATINGS

A.S. Kaygorodov[1,2*], A.S. Mamaev[1]

[1]Institute of Electrophysics UB RAS, Amundsen st. 106, Ekaterinburg, 620016, Russia

[2]Ural Federal University, Mira st. 19, Ekaterinburg, 620002, Russia

*e-mail: kaigor@iep.uran.ru

Abstract. A new experimental approach of TiC/a-C coatings critical relative indentation depth determination is presented. The data comparison of similar coatings obtained by magnetron sputtering on WC-based and stainless steel substrates shows that TiC/a-C nanohardness is affected by the substrate at indentation depths larger than 10-13 % of the coating's thickness. As for the friction coefficient, the soft substrate influences TiC/a-C coatings at the depths as small as 1 % of the coatings' thickness so this mechanical property is always influenced by the substrate.

1. Introduction

The coating of the surfaces for a wide range of applications is known for a long time. Initially the coatings were used generally for substrate protection. In this case, the dependence of the substrate's mechanical properties from the coating was studied. With the development of nanotechnologies the role of substrate changed from the main material to the carrier of nanostructured coating with unique properties. Here the influence of the substrate on the coating's properties has to be investigated. Several studies, both experimental and numerical ones, have analyzed the influence of substrates on the nanoindentation response of thin films [1-3]. The major objective of these investigations has been the simulation of hardness and modulus of the films, independent of the substrate, employing continuum analysis [4].

In common practice, it is generally assumed that a maximum indentation depth of one-tenth of the coating thickness should be sufficient to avoid the indentation curve being affected by the plastic deformation of the substrate [5, 6]. In case of a soft coating on a hard substrate the relative indentation depth can even exceed 30 % of the film thickness without the indentation curve being significantly influenced by the presence of the substrate since plastic deformation is generally confined within the coating. In contrast, when indenting a hard coating applied on a soft substrate the maximum indentation depth should be as small as 5 % of the coating thickness [7]. All attempts to evaluate the critical indentation depth by the numerical analyses refer to some generalized hard or soft coatings on the substrates regardless to the peculiarities of the specific coating material like chemical bonding, crystalline lattice, etc.

In present work, the experimental data on the influence of the substrate on the nanocomposite TiC/a-C coating's mechanical properties is analyzed. For this type of thin films the abovementioned experimental data is absent in the literature. For different amorphous carbon films on silicon substrate the critical indentation depth was found by the numerical analysis to be around 14 % of film thickness [8]. This value is much larger than 5 % obtained in [7], therefore this fact needs further clarification.

2. Materials and methods

In order to obtain TiC/a-C coatings the method of high-power pulsed magnetron sputtering (HPPMS) with titanium and carbon targets was used [9]. The resulting Ti/C ratio was 0.74-0.76. The targets were sputtered onto two substrates: hard WC-based alloy with the addition of T15K6-grade Ti (microhardness of ~17 GPa) and AISI430 stainless steel (microhardness of ~3 GPa). The surface of these substrates was polished with diamond pastes down to surface roughness of 15 and 30 nm respectively. The thickness of the coatings was varied from 150 to 2300 nm.

The surface roughness, R_a, was determined using Zygo NewView 5000 profilometer. The thickness of the deposited coatings, h, was measured using a Calotest device (CSM Instruments, Switzerland) by the ball surface abrasion method. The nanohardness of the coatings was measured by Nanotest 600 device (Micromaterial Ltd.) in the controlled depth mode with Berkovich diamond pyramid. The indenter penetration depth, d, was 100-1000 nm. In every measurement series the loading rate was varied to keep the loading time constant at the value of 20 seconds. The dwell time at the maximum load was 5 s. For analysis of the stress–strain curves, the Oliver and Pharr method [10] was used. The measurement results were averaged over a minimum of ten values in a set of 3 identical samples. For coefficient of friction determination a 55° cone indenter with 30 μm tip radius and different loading schedule were used. The scanning velocity of 5μm/s and scratch length of 1000 μm with linear loading up to 250 mN at 2.25 mN/s speed was suitable for friction force determination.

3. Results and discussion

The variation of indentation load allowed obtaining “depth-hardness” curves for both substrate types (Fig. 1). Here the elevated hardness at small indentation depth is explained by the indentation size effect [1] (curves 4 on Fig. 1a,b) and in case of AISI430 substrate by the additional influence of softer substrate on the hardness of the coating tending the resulting hardness to reach the hardness of the substrate at deep indents (curves 1-3 on Fig. 1b).

At the same time, a dependence of film nanohardness from its thickness is observed: the thicker the coating is indented the higher nanohardness at the same depths is reached. In case of hard WC substrate (Fig. 1a) this effect is very slight as almost all data lies in the error limits. The hardness of thick 900-2300 nm coatings starts from ~38 GPa at 100 nm depth and decreases down to ~20 GPa at 800 nm depth (curves 1, 2 on Fig. 1a). In case of soft AISI430 substrate this effect is more severe due to larger $H_{TiC}/H_{AISI430}$ ratio. The comparison of the coatings hardness at small depths around 100 nm (Fig. 1b) shows that the decrease of coating thickness from 2300 down to 150 nm leads to the decrease of its nanohardness from ~38 down to ~14 GPa. The main reason for that is the change of highly plastic substrate influence on the coating at different relative indentation depths. At deep indents the hardness of all samples tends to reach the hardness of pure substrate regardless of the thickness of the coating.

It can be seen that all the coatings curves on Fig. 1a are almost parallel to that of the substrate that is why it can be assumed roughly that the substrate does not affect the hardness of TiC/a-C due to close values of pure and coated substrate hardness. In this case the indentation curve for the coated WC substrate can be used as a reference one for the comparison with the data, obtained for other substrates with significantly different mechanical properties.

The influence of the substrate on the coating hardness can be clearly seen from the behavior of the indentation curves obtained for TiC/a-C coatings on hard and soft substrates (Fig. 2). Hard substrate does not influence the nanohardness of the coating here as the loading indentation curve is well fitted by Oliver and Pharr power equation (dashed part of curve 1). On the contrary the usage of the soft substrate changes the load-displacement curve

sufficiently (curve 2) so the fitting by the power law can be applied to the starting section of the loading curve only (dashed part of curve 2).

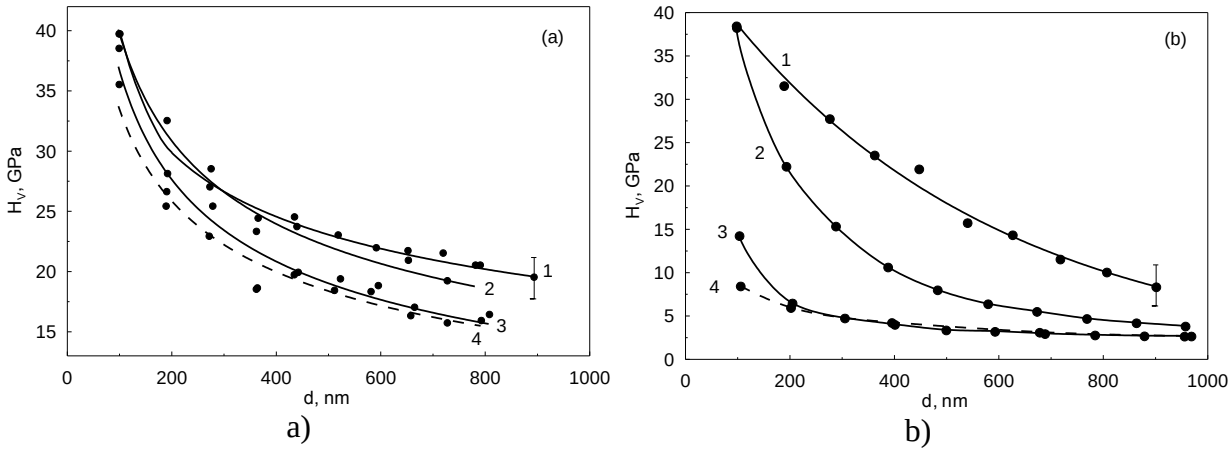

Fig. 1. The dependence of nanohardness from the penetration depth for TiC/a-C coatings on (a) WC and (b) AISI430 substrates. The numbers stand for the thickness of the coatings: 1-2300, 2-900, 3-150 nm, 4 – 0 nm (pure substrate). The data approximation of the coatings is given by solid lines, of the substrates – by dashed lines.

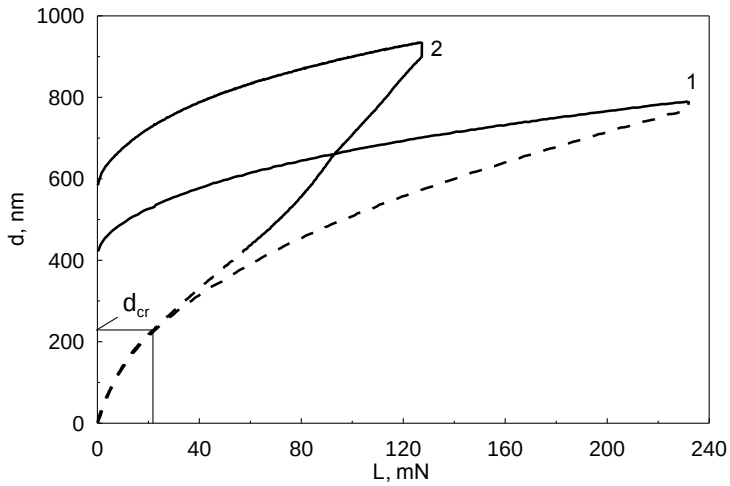

Fig. 2. The dependence of indentation depth from the applied to 2300 nm thick TiC/a-C coatings on WC (curve 1) and AISI430 (curve 2) substrates load.

For practical use, it is important to determine the critical indentation depth – the depth at which hardness becomes dependent from the substrate. In present work the critical depth, d_{cr}, was estimated as the point of divergence of the reference load-displacement curve (TiC/a-C on WC substrate) from TiC/a-C on steel substrate curve (curves 1 and 2 in Fig. 2) with determination error around 10 %.

As it is seen from Table 1 the critical relative indentation depth (CRID), given as d_{cr}/h, is depth sensitive with the magnitude of around 10-13 % for the wide range of coating thickness, h. This value is in good agreement with the general rule of so-called one-tenth of film thickness.

The same tendency of substrate influence can be observed for the coefficient of friction (Fig. 3). It is seen that the behavior of the coefficient of friction (CoF) for 2.3 μm thick TiC/a-C coating on AISI430 substrate sufficiently differs from that on hard WC substrate. The point of divergence for these two curves is located at around 8 mN. At this load the cone indenter is penetrated into the coating to the depth of 30 nm i.e. 1.3 % of the coating

thickness. It should be mentioned that such clear influence of the soft substrate can be observed for the thick coating only. In case of thinner coatings, the influence of the substrate appears to start at the lowest loads already and the point of divergence cannot be specified due to measurement error. Therefore, it can be stated that coating's coefficient of friction is sufficiently influenced by the substrate at any load applied.

Table 1. Critical indentation depth of TiC/a-C coatings on AISI430 substrate.

h, μm	d_{cr}, nm	CRID, %
0.5	63	13
0.9	105	12
2.3	225	10

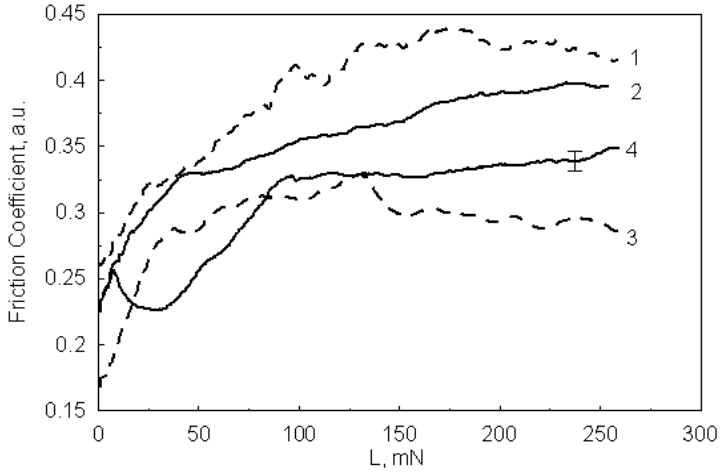

Fig. 3. Dependence of CoF from the load applied. 1 – pure WC substrate, 2 – 2.3 μm thick TiC/a-C coating on WC substrate, 3 – pure AISI430 substrate, 4 – 2.3 μm thick TiC/a-C coating on AISI430 substrate.

It is important to note that CRID value can be used here for evaluation purpose only. According to ISO 14577-1:2002 the indentation depth should be at least 20 times larger than the surface roughness, R_a, to eliminate its influence on the hardness measurements. Therefore, in terms of present experiment the minimal penetration depth for TiC/a-C coatings on AISI430 substrate should be around 300 nm and on WC substrate – 600 nm. Keeping in mind the value of CRID, derived in this work, the minimal thickness of the coatings should be not less than 3 and 6 μm for AISI430 and WC substrates correspondingly, but it appeared to be hard to synthesize such thick coatings with high adhesion by the abovementioned method. As the stable and homogeneous 2.3 μm thick TiC/a-C coatings obtained by magnetron sputtering had the best mechanical properties it was assumed that they can be used for CRID determination.

4. Conclusions

In present work, a new experimental approach for the critical relative indentation depth evaluation of the TiC/a-C coatings is proposed. The comparison of the data of the similar coatings obtained by magnetron sputtering on WC and stainless steel substrates shows that the indentation depths larger than 10-13 % of the coating's thickness lead to underestimation of TiC/a-C nanohardness on soft substrate. As for the friction coefficient it can be stated that the soft substrate influences TiC/a-C coatings at the depths as small as 1 % of the coating's thickness so this mechanical property is always affected by the substrate.

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