Mechanical Properties of Titanium Films Deposited by Pulsed High-Power Ion Beams

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Abstract – The adhesion of a titanium film to a silicon substrate, film nanohardness, Young modulus, and other properties have been investigated depending on a distance from a sputtered target to substrate and a thickness of the deposited film. Films were deposited from the ablation plasma formed under a pulsed high-power ion beam impinging on the titanium target. The analysis of experimental data has shown that at a given film thickness, the adhesion strength increases with increasing target-substrate distance; this dependence is stronger for thinner films. Adhesion significantly reduces as the film thickness increases, and this dependence is stronger at larger target-substrate distances. The friction coefficient of a diamond Rockwell indenter moving along the film considerably decreases, whereas the film nanohardness and Young modulus at a given indentation load grow as the target-substrate distance increases.

1. Introduction

There are a number of techniques being used to create thin-film coatings: physical and chemical vapor deposition, magnetron sputtering of deposited material, deposition from ablation plasma produced at a target under pulsed laser radiation. One of progressive methods is the pulsed ion beam deposition using high-power ion beams (HPIBs). It has a highest deposition rate, capable of preserving stoichiometry of deposited material, and requires lower energy expenses in comparison with other methods.

The important characteristic determining a quality of thin-film coatings is the adhesion. For metallic films, better adhesion can be achieved by maintaining an optimal temperature of a substrate during a film deposition [1, 2], applying a bias potential to a substrate [1, 3], ion and plasma processing of a substrate [4, 5], or creation of a transition layer between a film and a substrate [1, 6, 7]. When a film is deposited using a HPIB, the conditions of its formation change with varying distance from an ablated target to substrate, because the temperature of deposited plasma changes as well as film thickness produced per one pulse of the ion current. This was observed in the experiments performed at Sandia National Laboratories, USA [8]. Hence, the degree of the substrate heating is varied resulting in different film structure, mechanical characteristics, and adhesion to the substrate. In this work, the experimental results obtained at different target-substrate distances are presented for silicon substrates and titanium films of different thickness.

2. Experimental Procedure

For the creation of the ablation plasma and ion cleaning of the substrate surface, the HPIB of (70%H^+, 30%C^+) composition was employed. It was produced from the diode with spatial focusing installed at the accelerator "Temp-2". The accelerated ions energy was 350 keV, the ion current density ranged from 20 to 250 A/cm^2 corresponding to the beam energy density of 0.4–5.3 J/cm^2 at the HPIB pulse duration of 60 ns. The scheme of coatings deposition was presented in [2]. The target substrate distances were 55, 75, 90, and 190 mm. The thickness of films was measured by Linnik interferometer, the adhesion was estimated using the CSEM scratch-tester from the value of the critical indentation load, at which a film begins to flake away from the substrate, and from the acoustic emission signal. Also, the frictional force and friction coefficient were measured for the indentation load varied from 0.01 to 0.7 N. The CSEM nanohardness meter was used to determine films nanohardness, Young modulus, and loading-unloading curves.

3. Results and Discussion

The thickness of a film deposited per one pulse of the ion current is changed with changing target-substrate distance due to the angular divergence of the ablation plasma. In Fig. 1, the titanium film thickness dependences on the horizontal coordinate are presented for the three target-substrate distances *d*. It is seen that the profile of the deposited film thickness over the substrate can be approximately described by the Gaussian distribution. The film thickness deposited per one pulse significantly reduces (from 16.7 to 2.8 nm, i.e. ~6 times), as the target-substrate distance increases from 55 up to 190 mm, i.e. ~3.5 times. This reduction is conditioned by the divergence of the ablation plasma created under pulsed HPIB, which was measured in [9]. The actual form of the dependence of the thickness on *d* is affected by such factors as HPIB focusing in only one plane and plasma cooling during its motion from the target to the substrate.

In Fig. 2, the value of acoustic emission signal is shown (curve 2) for the load applied to the diamond indenter being varied from 0.01 to 2.5 N (curve 1).

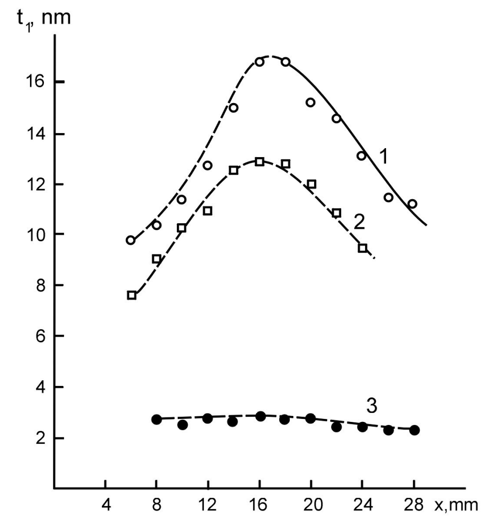

Fig. 1. Ti/Si film thickness (per HPIB pulse) profile at different target-substrate distances $d = 55$ (1); 90 (2); 190 mm (3). x is the horizontal coordinate

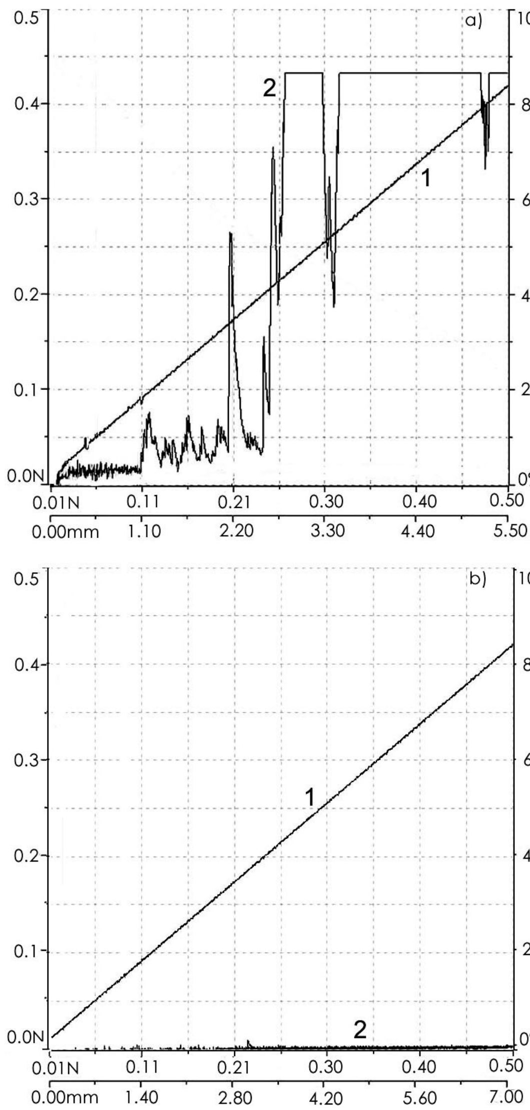

Fig. 2. Acoustic signal (2) versus indentation load (1). The thickness of Ti/Si film is: a – 0.47 μm; b – 0.09 μm. $d = 90$ mm.

The film thickness is: a) $t = 0.47$ μm, b) $t = 0.09$ μm; in both cases, the distance $d = 90$ mm. The critical load, at which the film of 0.47 μm thickness begins to flake away, $F_{cr} = 0.1$ N. For the film of $t = 0.09$ μm, no exfoliation was observed within this range of the indentation load; in this case, $F_{cr} = 0.48$ N. For the film of intermediate thickness $t = 0.26$ μm, the critical load $F_{cr} = 0.137$ N. Thus, as the film thickness reduces, its adhesion increases significantly. The similar dependences were observed also at other distances between the target and substrate.

The values of the critical load for the films of approximately the same thickness obtained at different target-substrate distances (that was achieved by using a corresponding number of HPIB pulses in each case) are presented in the Table 1. It is seen that the adhesion significantly depends on the distance d. Increasing d results in the reduction of not only the thickness of the plasma layer deposited per one HPIB pulse, but also the plasma temperature, so that conditions of the film formation at the substrate become different. For smaller temperature difference between the hot plasma and cold substrate, the difference between the linear expansion coefficients of the film and substrate produces a smaller effect of occurrence of internal tensions in the deposited film [6], which leads to the reduction of adhesion. Another cause of adhesion improving with increasing target-substrate distance is the substantial decrease of the number and sizes of microdroplets. In the work [10], the pulsed ion beam deposition was made at target-substrate distance of 30–50 mm, and the presence of microdroplets and bubbles in metallic films was reported, whose sizes reached hundreds nanometers, and volume concentration was several percents. The microphotographs of the films obtained in our experiments show that the portion of microdroplets dramatically reduces as the target-substrate distance increases.

Table 1. Critical indentation load F_{cr} for films obtained at different target-substrate distances d

d, mm	55	75	190
Film thickness t, μm	0.15	0.13	0.14
F_{cr}, N	0.1	0.38	> 0.5

In Fig. 3, the dependences of the frictional force and friction coefficient on the load applied to the indenter are shown for the films deposited at different target-substrate distances. For the films of comparable thickness ($t = 0.15$ μm and $t = 0.14$ μm), the significant difference in the friction coefficient is observed.

The important mechanical characteristics of coatings are also their hardness and elasticity. The measured values of nanohardness (by Vickers) and Young modulus for Ti/Si coatings obtained at different distances d are presented in the Table 2. The indentation load was 2 mN. In Fig. 4, the loading and unloading curves for these coatings are shown. With increasing target-substrate distance, the coating nanohardness substantially increases, that means films turn out denser, i.e., of higher quality. At the same time, the plasticity increases as well; this may be both advantageous (in the main) and disadvantageous property of a film.

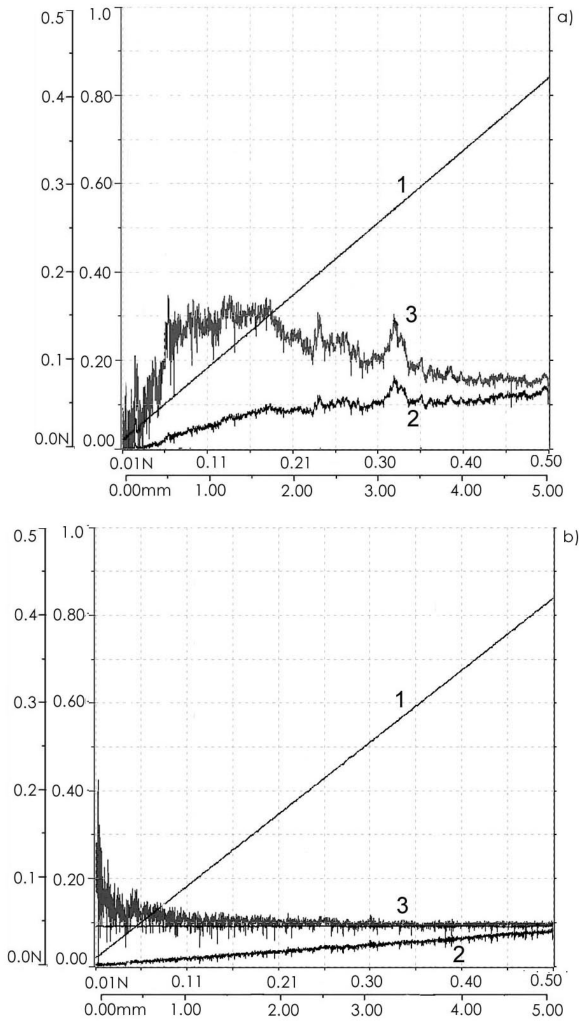

Fig. 3. Frictional force (2) and friction coefficient (3) versus indentation load (1). a – $t = 0.15$ μm, $d = 55$ mm; b – $t = 0.14$ μm, $d = 190$ mm

Table 2. Nanohardness H_v and Young modulus E for Ti/Si coatings obtained at different target-substrate distances d

d, mm	55	75	90
Film thickness t, μm	0.23	0.13	0.068
Depth of indenter penetration, nm	120	110	58
H_v, GPa	6.51	7.15	55.15
E, GPa	86.1	123.8	318.1

4. Conclusion

The investigations performed have shown that the deposition rate of titanium films decreases with increasing target-substrate distance. This dependence is much stronger than that observed in the experiments at SNL [8], however, it is weaker than $\sim d^{-2}$, which is typical for laser or vacuum arc deposition. The adhesion of Ti/Si coatings substantially reduces as their thickness increases and the target-substrate distance decreases. The friction coefficient decreases, and nanohardness and Young modulus increase with increasing target-substrate distance.

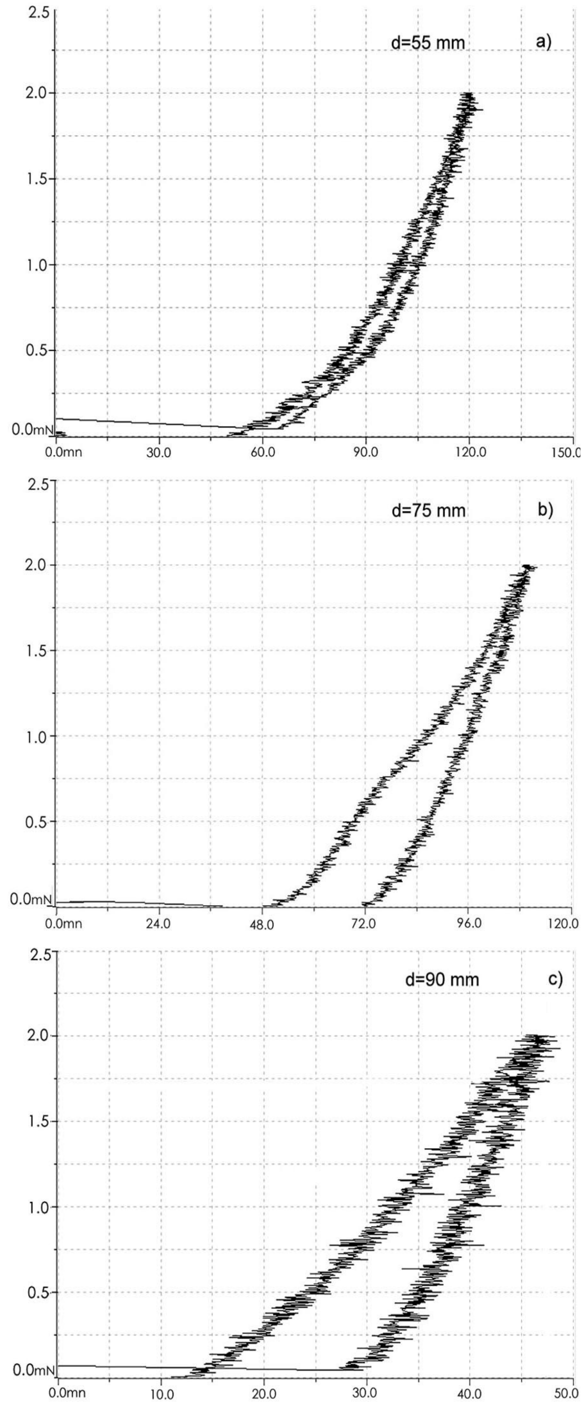

Fig. 4. Loading and unloading curves for Ti/Si coatings deposited at different distances d

Thus, the distance between the ablated target and substrate significantly influences on the adhesion and mechanical characteristics of thin-film coatings deposited using pulsed HPIBs. For the given ion beam energy density (~ 4.2 J/cm^2), high-quality films can be obtained for the distances $d \geq 90$ mm.

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