

Mechanical properties of titanium nitride films obtained by reactively sputtering with hot target

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Abstract. The mechanical properties and morphology of the surface of titanium nitride films deposited by reactive sputtering with a hot target have been investigated. It was founded that films synthesized at low nitrogen flow rate and at high discharge current density have the highest hardness (up to 30 GPa) and Young's modulus (up to 300 GPa). The analysis of films surface images obtained by atomic force microscopy is carried out. The typical grain size and its shape were determined. Films with the greatest hardness have the largest grains.

Titanium nitride (TiN) retains leading position among transition metals nitrides that can be synthesized as thin films. TiN films have high electrical conductivity, adhesion, hardness and chemical inertness therefore they are used as decorative, wear-resistant and corrosion-resistant coatings, for modifying the metal surface against fretting corrosion. Also they are applied in silicon technology as diffusion barriers for aluminum and copper metallization, when creating MOS transistors, ohmic and rectifying contacts. In the recent time high power reactive magnetron sputtering (sometimes with hot target) has become the most popular method of obtaining titanium nitride thin films.

In the present work research of titanium nitride films deposited by DC reactive sputtering of a hot target in a mixture of argon and nitrogen gas have been carry out. The films were made in a vacuum chamber with a volume of $7.8 \cdot 10^{-2} \text{ m}^3$ equipped with a planar magnetron with a titanium target 130 mm in diameter. The hot target regime is provided by fixation a 1 mm thick titanium disk with a gap of 1 mm on a 4 mm thick water-cooled copper plate. The residual pressure in the chamber did not exceed 10^{-2} mTorr . Sputtering was carried out in Ar/N₂ at a pressure $p_{\text{Ar}} = 2 \text{ mTorr}$, nitrogen flow rate 2–6 cm³/min, and discharge current density 14–33 mA/cm².

The mechanical properties (hardness and modulus of normal elasticity) were measured by the nanoindentation using the NanoScan-4D nanohardness measuring [1–3] (FGBN TSUM, Russia). This method allows studying evolution processes of both elastic and plastic deformation in small volumes. The Berkovich indenter in the shape of triangular diamond pyramid with an apex angle of about 142° was used for measurements. In nanoindentation method the indenter is pressed into the surface of the sample at a set rate with registration of the applied load and the corresponding displacement of the indenter.



The study of surface morphology was performed by atomic-force microscopy (AFM) using the INTEGRA Prima atomic force microscope (NT-MDT, Russia) in the semi-contact mode using silicon cantilevers with a needle radius of 10 nm (NSG01 series) (figure 1). The scanning speed did not exceed 0.5 $\mu\text{m/s}$. Image analysis is performed using the Nova software (NT-MDT).

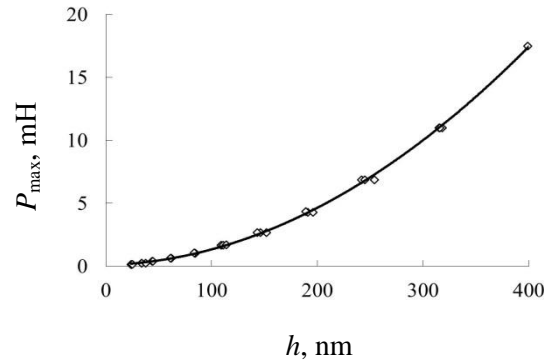


Figure 1. Loading diagram of the sample.

Figure 1 shows a typical loading diagram of the samples in our studies. The diagram is shown in the coordinate system “load P vs. depth of the indenter penetration h ” with a maximum load of about 20 mN. Based on experimental results, for each sample nano-hardness and Young’s modulus were calculated. The results of the calculations are shown in figure 2.

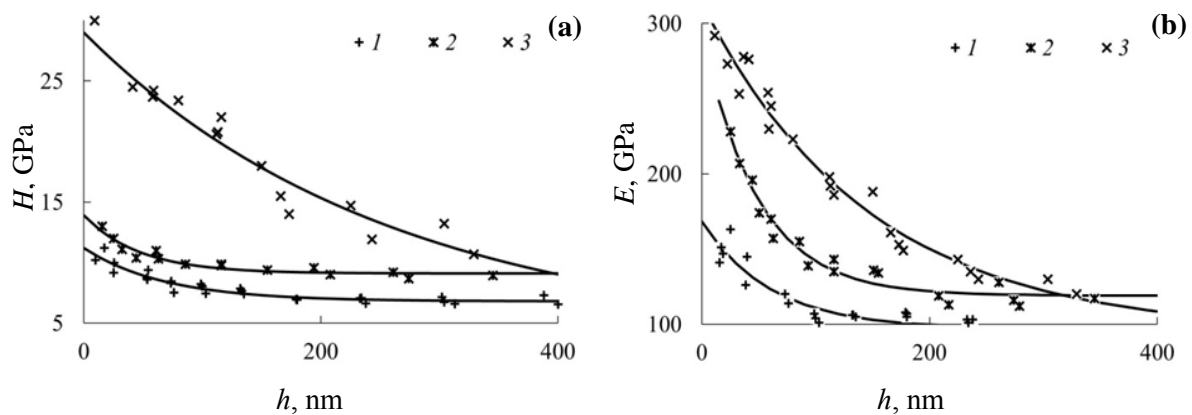


Figure 2. Nanohardness (a) and elastic modulus (b) of films produced with nitrogen gas flow of 2 cm^3/min and current density (mA/cm^2) of: 1 – 14; 2 – 22; 3 – 33. Dots are for experimental results of indentation with penetration depth h , lines are approximations.

It can be seen from figure 2 that the highest hardness (around 30 GPa) and Young’s modulus (around 300 GPa) are possessed by films deposited with the highest current density. The ones deposited with the lowest current density have surface properties that insignificantly vary from the substrate surface properties. For glass substrates, the values of H and E are equal to 7 and 80 GPa respectively.

The surface morphology studies were performed by AFM. For results processing we used a specialized Nova software that allows accurate surface visualization and perform a film characterization by various statistical methods.

Figure 3 shows typical 3D images of films deposited with constant nitrogen inlet flux. Images were modified by using a line-by-line filtering for elimination of inclined plane effect occurred due to impossibility of placing a sample exactly perpendicular to atomic-force microscope cantilever.

A root mean square (RMS) roughness was chosen as a parameter for characterizing film surface. By using Nova software tool “Roughness analysis” it was found that with increasing of gas flow from 2 to 6 cm^3/min the RMS roughness decreases from 1.5 to 1.3 nm at $j = 13.7 \text{ mA}/\text{cm}^2$ and from 6.1 to

3.3 nm at $j = 32.9 \text{ mA/cm}^2$. This decreasing of RMS roughness with increasing of gas inlet is in good correlation with data for titanium nitride films obtained by reactive magnetron sputtering studied by other authors [4].

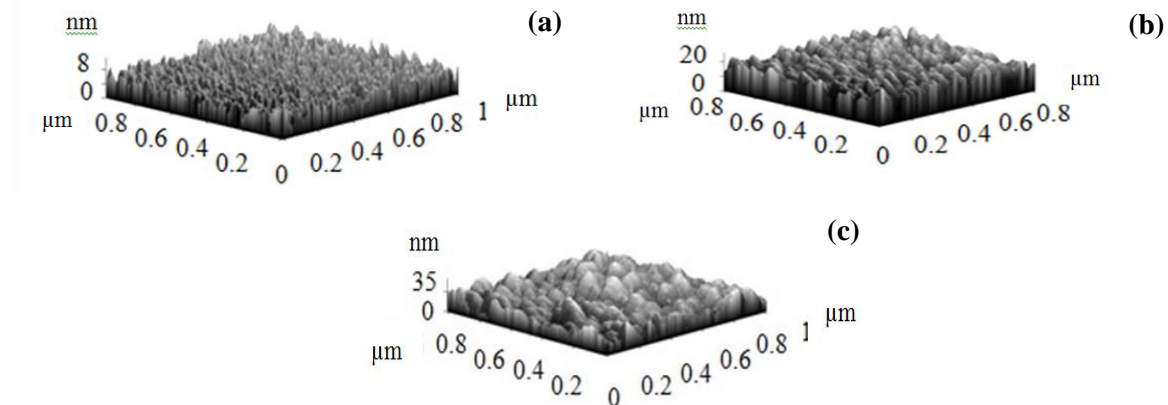


Figure 3. AFM images of films produced with current density (mA/cm^2): (a) –14; (b) – 22; (c) – 33.

Analysis of film grains was performed using “Grain analysis” tool. The principle of the method is as follows: user cut the sample with a plane parallel to the substrate and placed on a given distance from it. It was defined that at constant gas flow rate of $2 \text{ cm}^3/\text{min}$ with increasing of current density from 14 to 33 mA/cm^2 , a typical size of grains increases from 50 to 70 nm. At the same time, a shape of grains becomes flat-topped. At the constant current density of 14 mA/cm^2 , increasing of gas flow rate from 2 to $6 \text{ cm}^3/\text{min}$ cause not only decreasing of a typical grain size to 20 nm but also a more needle-like form of grains.

The proposed method can be used in the complex study of surface morphology as it per-form to make a conclusion about a main mechanism of crystallite growth in a film. The titanium nitride films have been deposited by DC reactive sputtering of a hot target in a mixture of argon and nitrogen gas.

Comparing the results of film morphology and its micro-hardness studies it can be said that the increasing of crystallites typical size correlates with the increasing of film micro-hardness. The sample having the largest crystallites exhibit the most hardness as well. This sample was obtained under conditions of the most current density and the least gas flow rate i.e. the most deposition rate.

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