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DEPOSITION TEMPERATURE INFLUENCE ON THE MECHANICAL PROPERTIES OF NIOBIUM NITRIDE THIN FILMS

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Abstract: *Niobium nitride is used in different applications due to its mechanical, tribological, electrical, and optical properties. This paper is a study concerning the influence of the deposition temperature on the mechanical characteristics of niobium nitride thin films at nanoscale. The films were deposited on silicon substrates by direct current magnetron sputtering at different temperatures. The deposition was done at room temperature, at 200 and 400 °C respectively. Atomic force microscopy investigations were performed to determine the mechanical properties of the niobium nitride films. The results pointed out that both the nanohardness and modulus of elasticity are decreasing with the increase in temperature up to 200 °C after which they are increasing when the temperature is increased up to 400 °C.* ***Key words:*** *niobium nitride, magnetron sputtering, nanoindentation, nanohardness, modulus of elasticity.*

1. INTRODUCTION

The deposition of nitride thin films by magnetron sputtering, as well as their characterization find their applicability in several engineering industries due to their properties such as high hardness, high melting point, good corrosion resistance, good superconducting properties etc. [1-3]. One of the industries that presents direct interest for the current paper is the manufacturing of MEMS (microelectromechanical systems) devices. On these lines, the niobium nitride can be used as diffusion barrier [4].

Scientific research conducted in the last years show an interest towards developing devices that are characterized not only by increased efficiency, but also by reduced dimensions (micro- (MEMS) or even nano-systems (NEMS)). The industry dedicated to developing and manufacturing such devices has grown like never before. Now each device meets a certain need of a certain client, fact that requires to manufacture the device considering aspects such as material choice or elaboration technology.

The exact determination of mechanical and tribological thin films used to manufacture MEMS devices is a must at micro scale as well as nano scale due to the fact that their functioning is strongly influenced by the characteristics of the materials used to manufacture them. The optimal functioning of MEMS devices is obtained when the efforts focus also on determining the mechanical properties of the MEMS materials [5].

Niobium nitride thin films present interest to the researchers nowadays due to their applicability as superconductors [6] and as well as diffusion barriers [7]. In the scientific literature, several such thin films obtained through different methods have been studied [7-10]. The results obtained after the investigations were performed show the need to control the deposition process of the thin films in order to obtain certain textures that allow to obtain some superior mechanical and tribological properties. Atomic force microscopy tests can be performed on such materials in order to determine their properties and to understand the processes that take place at their surface.

The present paper encompasses the research regarding the influence of the deposition temperature on the mechanical properties – namely nanohardness and modulus of elasticity

– of niobium nitride thin films deposited by direct current (DC) magnetron sputtering on silicon substrates.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

The deposition of the niobium nitride thin films was done by DC magnetron sputtering using a niobium target with the purity of 99.95 %. Silicon Si (100) surfaces were employed as substrates. The process took place in a chamber of a reactive sputtering facility with high vacuum of 10^{-7} torr, containing a mixture of argon and nitrogen. The following parameters were insured: discharge current of 350 mA, argon flow rate of 40 $cm^3 \cdot min^{-1}$, nitrogen flow rate of 1.5 $cm^3 \cdot min^{-1}$, pressure inside the chamber of 2 mtorr. The distance between the niobium target and the silicon substrates was kept constant at 60 mm. First the substrates were cleaned in an ultrasonic bath using isopropyl alcohol to remove any possible impurity. The deposition time was 20 minutes. The thickness of the deposited films was determined to be about 0.35 µm. The deposition took place at different temperatures. Some films were deposited at room temperature, other films were deposited at 250 ºC and the rest of the films were deposited at 400 ºC.

Once the samples were deposited, they were characterized by atomic force microscopy investigations. The tests were performed using a XE 70 atomic force microscope (AFM) from the Micro and Nano Systems Laboratory. The testing temperature was 20 °C and the relative humidity was 30 %. The nanoindentation tests performed to determine the mechanical properties were done using a TD 21464 nanoindentor. As the manufacturer indicated, the characteristics of this indentor are: cantilever stiffness of 156 $N \cdot m^{-1}$, its length of 581 µm, tip thickness of 19 µm, tip height of 103 µm and tip radius smaller than 25 nm. The tests were performed for a force limit of 20 nN. Once the Z scan vs. force curves were obtained, they were interpreted with the XEI Image Processing Tool for SPM (Scanning Probe Microscopy) data using both the Oliver and Pharr and the Hertzian.

3. RESULTS AND DISCUSSION

Our first priority was to investigate the surface of the deposited niobium nitride thin films.

3.1 Topography

Each sample was scanned using the XE 70 atomic force microscope. The XEI Image Processing Tool for SPM data allowed us to obtain the 3D images of the surfaces for each film. Fig. 1 presents a 3D image of a niobium nitride thin film deposited at 20 °C.

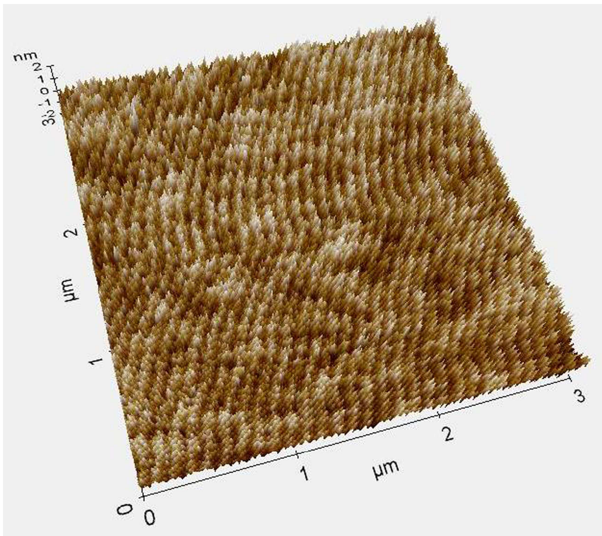

Fig. 1. 3D image of a niobium nitride thin film deposited at 20 °C.

The software also allowed us to determine the average roughness of the investigated films. An image of the XEI software used when determining the roughness parameter is presented in Fig. 2. The values obtained for the average roughness are given in Table 1.

Table 1

Average roughness of the deposited niobium nitride thin films.

Deposition temperature (°C)	**20**	**200**	**400**
Average roughness (nm)	0.386	0.592	0.677

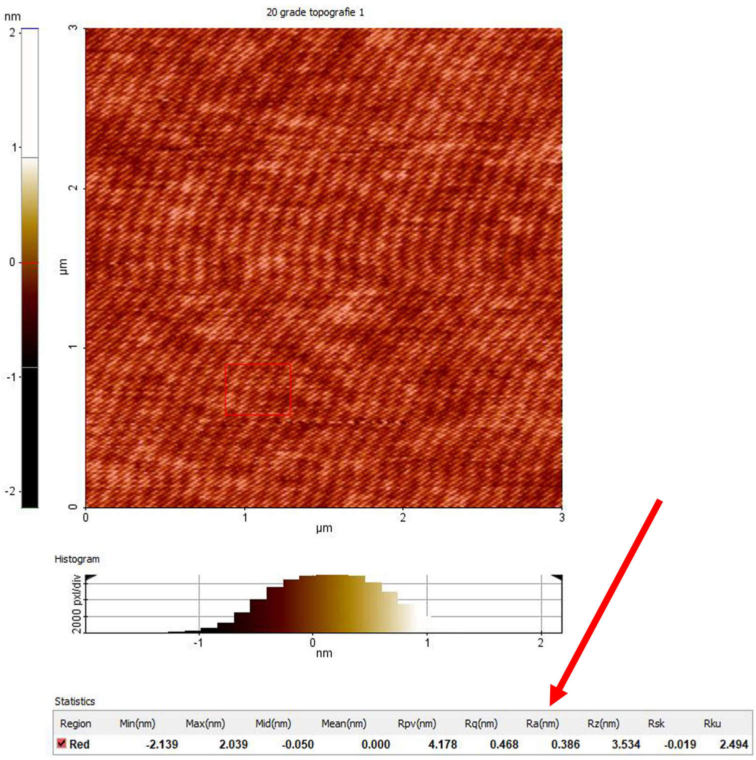

Fig. 2. Image of the XEI Image Processing Tools for SPM Data for determining the average roughness of a niobium nitride thin film deposited at 20 °C.

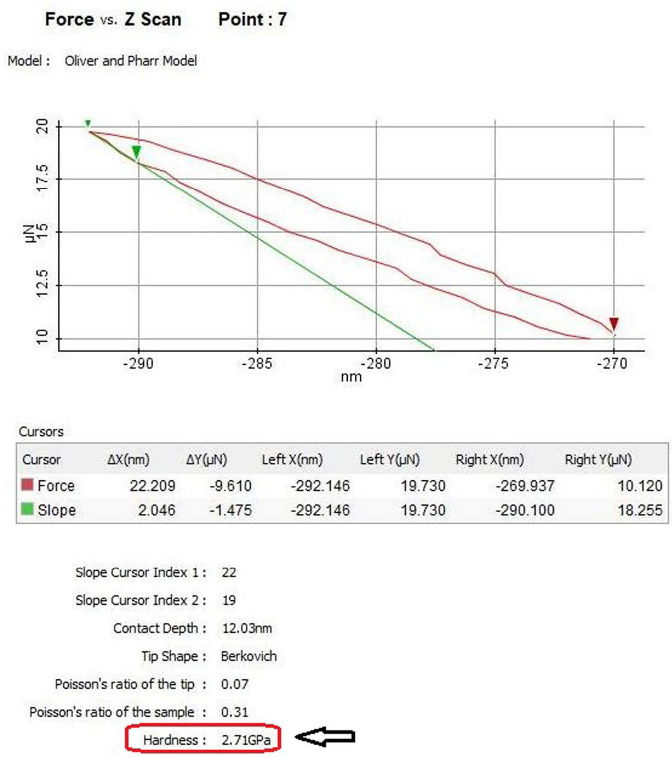

Fig. 3. Image of the XEI Image Processing Tools for SPM Data for determining the nanohardness of a niobium nitride thin film deposited at 20 °C.

It is noted that once the deposition temperature increases from room temperature up to 400 °C, the average roughness increases as well. If when we increased the temperature from 20 to 200 °C, the roughness increases more than 53 %, in the case when we further increased the temperature up to 400 °C, the increase was much smaller (about 14 %).

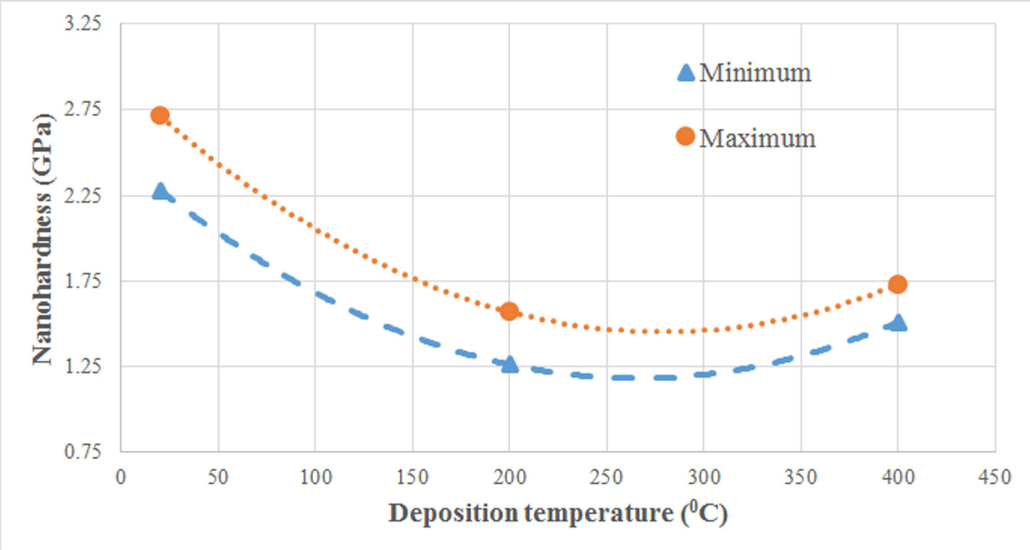

Fig. 4. Fluctuation of the nanohardness according to deposition temperature for the deposited niobium nitride thin films.

3.2 Nanohardness

The mechanical characterization implied the determination of the nanohardness and the modulus of elasticity of each film. After the nanoindentation tests, we've obtained the Z scan vs. force curves specific to each type of deposited sample. The Oliver and Pharr method was employed for determining the nanohardness. This method assumes that during the tests plastic deformation occurs while the Hertzian method assumes that there only elastic deformation occurs at nanoindentation. Fig. 3 presents an image of the XEI software when interpreting a Z scan vs. force curve of a niobium nitride thin film deposited at 20 °C for determining the nanohardness. The fluctuation of the nanohardness according to the deposition temperature is graphically given in Fig. 4. Sixteen tests were performed. The minimum and the maximum values obtained for each type of sample are given on the graph. One can see that the temperature has a negative effect on the nanohardness. Once we increase the temperature up to 200 °C, the nanohardness decreases strongly with about 43 %. If the average value of the nanohardness for the films deposited at 20 °C is about 2.5 GPa, the average value of the nanohardness for the films deposited at 200 °C is about 1.42 GPa. The subsequent increase in temperature up to 400 °C leads to an increase of the nanohardness of about 14 %. The average value of the nanohardness for the films deposited at 4200 °C is 1.62 GPa. However, it should be noted that the films deposited at 400 °C present a narrower range than the films deposited at room temperature or at 200 °C.

3.3 Modulus of elasticity

The second mechanical characteristic of interest is the modulus of elasticity. The determination of this property can be achieved using the Hertzian method for interpreting the Z scan vs. force curves. Fig. 5 presents the variation of this mechanical characteristic in terms of deposition temperature.

A similar trend to those specific to the nanohardness can be observed even in the case of the modulus of elasticity. Once again the property is ranged between a minimum and a maximum value that were determined after testing in sixteen points. The niobium nitride thin films deposited at 20 °C show a narrow range in comparison with the films deposited at 200 or 400 °C. The films deposited at room temperature show a modulus of elasticity of about 113 GPa, higher than the films deposited at 200 °C with about 34 %.