Analysis of humidity influence on adhesion and tribological properties of niobium nitride thin films

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Abstract. This paper is focused on characterizing several samples of niobium nitride thin films using an atomic force microscope at different humidity levels. The samples were deposited by direct current reactive magnetron sputtering on silicon substrates by varying the deposition temperature from room temperature up to 400 °C. The main objective of this paper is to investigate the influence of the humidity level on the adhesion force and on the tribological properties for this type of thin films. Hence, the relative humidity was ranged between 30 % and 80 % and the adhesion force between the AFM tip and each sample was experimentally determined using the spectroscopy in point mode of the AFM. Also, the friction force was determined, and then, based on its experimental values as well as on the values obtained for the adhesion force, the friction coefficient between the AFM tip and the sample was computed. The results obtained from the conducted experimental investigations allow establishing the niobium nitride thin films which have superior properties with respect to adhesion and tribology.

1. Introduction

Thin films science and technology are the centre of many researchers work due to their significant part in the high-tech industries. Their use in manufacturing of micro-electro-mechanical systems (MEMS) as superconductors and as diffusion barriers ([1,2]) have led to intense research on thin film elaboration and characterization ([3-5]).

The research conducted in the last decade shows investigations regarding the influence of fabrication parameters on the structure of NbN thin films [6,7]. However, an approach that combines the influence of the deposition parameters with the influence of the operating conditions has not been reported yet. Moreover, the reported adhesion investigations were conducted using scratch tests [8]. In contrast, the current paper encompasses the research regarding the influence of the relative humidity on the adhesion and friction force of NbN thin films deposited at different temperatures while the experimental investigations were conducted using atomic force microscopy (AFM).

2. Materials and experimental procedure

2.1. Materials

This paper is focused on investigating the humidity influence on adhesion of three samples of niobium nitride thin films. The samples were obtained by depositing one layer of niobium nitride on a silicon Si (100) substrate by reactive magnetron sputtering using a Nb target with a purity of 99.95%.
The deposition took place in a high vacuum chamber (10^{-7} \text{ torr}) containing a mixture of argon and nitrogen. All deposition parameters except the temperature were kept constant and all samples were deposited for the same period of time. As shown in table 1, the deposition temperature was varied between 20 °C and 400 °C.

### Table 1. Deposition conditions for investigated samples.

| Sample   | T (°C) | Time (min) | P (mtorr) | Id (mA) | Q_{Ar} (cm^3/min) | Q_{N2} (cm^3/min) |
|----------|--------|------------|-----------|---------|-------------------|-------------------|
| Sample_1 | 20     | 20         | 2.2       | 300     | 40                | 1.5               |
| Sample_2 | 250    | 20         | 2.2       | 300     | 40                | 1.5               |
| Sample_3 | 400    | 20         | 2.2       | 300     | 40                | 1.5               |

2.2. Experimental procedure

The characterization of the deposited samples was conducted by atomic force microscopy investigations. The tests were performed using the XE 70 AFM from the Micro and Nano Systems Laboratory from the Technical University of Cluj-Napoca. The testing temperature was kept constant (ambient temperature) while the relative humidity was varied between 20 % and 80 %. The cantilever used for these tests is a PPP-NCHR 10M cantilever.

First, the samples were characterized from the topographical point of view by scanning each sample surface at 20 %RH with the AFM cantilever in contact mode. The scanning also allowed the tribological characterization of the samples.

Secondly, the samples were characterized from the adhesion point of view by performing spectroscopy in point mode of the AFM. The technique consists in bringing the tip of the AFM cantilever in the proximity of the sample surface. Then, the phenomenon known as “snap-in” occurs (they come in contact) due to the existing attraction forces. The loading continues and the AFM cantilever is deflected. When the cantilever retracts to its initial position (during the unloading) the pull-off phenomenon occurs (the contact between the tip and the sample is broken). Using this technique, AFM experimental force vs. z scan curves are obtained for all samples tested at different values of the relative humidity (from 20 %RH to 80 %RH with a step of 20 %RH). The experiments conducted in order to determine the adhesion were repeated three times in three different points of each sample surface and the XEI Image Processing Tool for SPM (Scanning Probe Microscopy) Data was used for data interpretation.

3. Results and discussions

The first type of investigation consisted in scanning each sample in contact mode in order to obtain the topography. The 3D images of each thin film were provided by the data interpretation software (see for example figure 1). Each sample was also scanned at different relative humidity levels in order to determine its influence on the friction force. First, the deflection of the tip was determined and then, based on its value the friction force was computed. The results are presented in table 2.

The second type of investigation was the spectroscopy in point mode. Each sample was tested at four different levels of relative humidity (20 %RH, 40 %RH, 60 %RH and 80 %RH) and in three random points of the sample surface. The points were chosen by analysing the 3D image of each sample in order to avoid any defects. The XEI Image Processing Tool for SPM Data provides values for other characteristics besides the adhesion (pull-off) force as it can be seen in figure 2.

After experimentally determining the value of the adhesion force for each sample in three random points at all tested relative humidity levels, the average value of the adhesion force was computed and the results are presented in table 3. It can be observed that for all samples the adhesion force first has an increasing trend up to a maximum value, then followed by a decrease when compared to the increase of the relative humidity. The reference sample deposited at 20 °C has the highest value of the adhesion force and it is obtained at 60 %RH. The other two samples deposited at higher temperatures
than ambient temperature have lower adhesion force values for all relative humidity levels and they reach the maximum value at 40 %RH.

Table 2. Friction force values for all samples at different levels of relative humidity.

| Sample    | 20 %RH | 40 %RH | 60 %RH | 80 %RH |
|-----------|--------|--------|--------|--------|
| Sample_1  | 82.29  | 245.30 | 327.41 | 210.67 |
| Sample_2  | 116.73 | 509.55 | 251.56 | 142.83 |
| Sample_3  | 74.81  | 435.27 | 214.85 | 172.92 |

Figure 1. The 3D images of the thin films for Sample_1.

Figure 2. The force vs. z scan curve together with the characteristics provided by the AFM software for the third experimental test conducted on the Sample_1.
Table 3. Adhesion force values for all samples at different levels of relative humidity.

| Sample    | 20 %RH | 40 %RH | 60 %RH | 80 %RH |
|-----------|--------|--------|--------|--------|
| Sample_1  | 116.30 | 637.00 | 767.50 | 374.60 |
| Sample_2  | 87.10  | 538.00 | 227.69 | 127.35 |
| Sample_3  | 109.20 | 632.41 | 292.17 | 163.00 |

4. Conclusions
This paper is focused on determining the humidity influence on the adhesion force as well as the friction force for different samples of niobium nitride thin films. For both studied characteristics the increase of the relative humidity first determines an increase followed by a decrease of the values for all samples. At a level higher that 60 %RH both friction and adhesion forces are higher for the Sample_1. The maximum values for both studied forces is obtained at a 60 %RH for the reference sample deposited at 20 °C and at a 40 %RH for the samples deposited at higher temperatures than the ambient temperature.

The friction force for Sample_1 and Sample_2 has a maximum value approximately four times higher than the one at a 20 %RH while for the Sample_3 it is almost six times higher than the one obtained at 20 %RH. The adhesion force for the Sample_1 has a maximum value approximately 6.5 times higher than the one at a 20 %RH while for the Sample_2 and the Sample_3 it is almost six times higher than the one obtained at 20 %RH. Also, it can be concluded that the increase of the deposition temperature determines a decrease of the adhesion force values regardless of the relative humidity level at which the tests are conducted.

5. References
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