Effect of argon ions implantation on the reactively sputtered TiN layers

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Thin films of TiN have unique combinations of properties such as high melting point, high hardness, low resistivity, good chemical stability, high wear resistance, and high reflectivity in the infrared region [1]. The aim of the present work is to study the changes of the micro-structural properties of TiN thin films induced by Ar ions implantation.

Thin films of TiN were deposited on Si(100) substrates by reactive ion sputtering in a Balzers Sputtron II system. The base pressure in the chamber was around $1x10^{-6}$ mbar, the argon partial pressure was $1x10^{-3}$ mbar while the nitrogen partial pressure was $3x10^{-4}$ mbar. During deposition, the substrate temperature was held at room temperature (RT) and at 150^{o}C. The TiN layers were grown at a rate of~8nm/min, to a total thickness of ~240 nm as measured with a profilometer. Samples were then irradiated at room temperature with 120 keV Ar^{+} ions to the fluences of $1x10^{15}$ and $1x10^{16}$ ions/cm^{2}. Structural characterization of the as-deposited and irradiated samples was performed with Rutherford backscattering spectrometry (RBS), transmission electron microscopy (TEM) and x-ray diffraction (XRD). For RBS we used 900 keV He^{++} ion beam at normal incidence with a detector positioned at a scattering angle of 165^{o}. All the samples were analyzed by x-ray diffraction (XRD) at 1^{o} incidence, using CuK_{α} emission. The mean grain size of the TiN thin films was estimated from the Scherrer's formula [2]. The microstructure of the TiN thin films was also analyzed by transmission electron microscopy (TEM), using Philips EM 400 microscope operated at 120 keV. We used micro-diffraction (MD) technique to study the crystalline structure of the TiN layers. Cross-sectional TEM samples were first mechanically polished and than were ion milled.

Fig 1 illustrates the Ti, N, Si and Ar (incorporated during the sputter deposition) depth profiles in a sample deposited at RT. The depth profiles of the layer components were extracted from the experimental RBS spectra by means of the WiNDF code. The profiles show a nearly uniform stoichiometry of the TiN layer and 1-2 at % of Ar. We have presented the depth profiles taken from the sample deposited at RT, although similar extracted profiles are also observed at 150^{o}C, and after irradiation with 120 keV argon ions.

The results show that the mean grain size decreases from ~13nm to ~7nm when increasing ion fluences. It is clear that the observed changes are influenced by ion irradiation, which induces local structural rearrengements and defects formation. The mean grain size slightly increases to ~16 nm when increasing temperature (150^{o}C) during the deposition. The atomic species have higher mobility at 150^{o}C and consequently the larger crystallites are formed.

Cross sectional TEM analysis revealed that the as deposited layers grow in a form of a polycrystalline columnar structure which was shown in our previous paper [3]. A typical set of micro-diffraction (MD) patterns is given in Fig 2 (a-c). The MD patterns were taken from an as-deposited sample at RT (a), from a sample implanted to $1x10^{16}$ ions/cm^{2} (b), and from a sample deposited at 150^{o}C(c). The diffraction spots along the (111), (200), (220) and (311) rings are indication of very fine polycrystalline structure. The MD pattern in (b) indicates that the polycrystalline structure is retained after ion implantation. However, a slightly finer grain

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structure is observed. Besides, the microstructure of the layer deposited at 150°C, consists of slightly larger crystalline grains, as shown in (c).

In conclusion, we have shown that the microstructure and of TiN layers depend on the deposition temperature and Ar ions irradiation. The crystalline grain size slightly increases, when increasing the temperature (150°C) during deposition process. After irradiation the microstructure of the TiN layers remains polycrystalline, but the mean grain size decreases.

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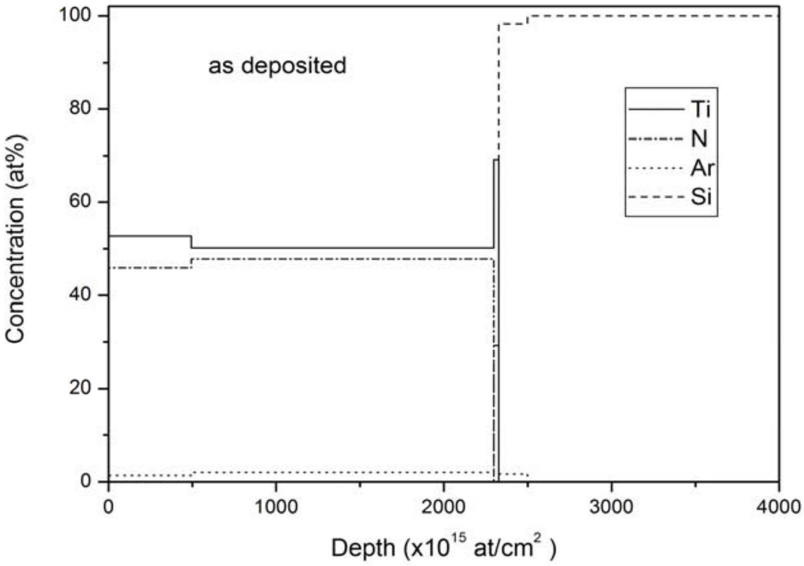

Figure 1. Extracted depth profiles from the sample deposited at RT

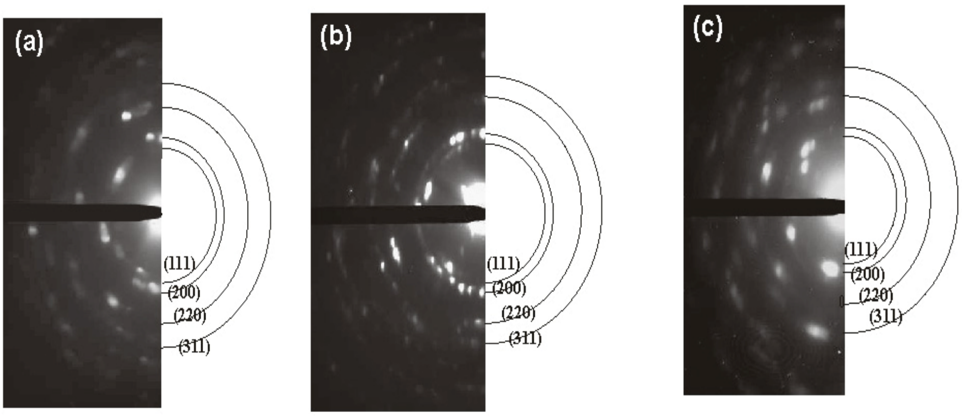

Figure 3. MD patterns of TiN sample: (a) deposited at RT; (b) deposited at RT and irradiated to 1x10^{16} Ar/cm^2; (c) deposited at 150°C.