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Reactively sputtered Ni, Ni(N) and Ni_3N films: Structural, electrical and magnetic properties

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ABSTRACT

Nickel thin films were deposited on glass substrates at different N_2 gas contents using a dc triode sputtering deposition system. Triode configuration was used to deposit nanostructured thin films with preferred orientation at lower gas pressure and at lower substrate temperature compared to the dc diode sputtering system. A gradual evolution in the composition of the films from Ni, Ni(N), to Ni_3N was found by X-ray diffraction analysis. The preferred growth orientation of the nanostructured Ni films changed from (1 1 1) to (1 0 0) for 9% N_2 at 100 °C. Ni_3N films were formed at 23% N_2 with a particle size of about 65 nm, while for 0% and 9% of nitrogen, the particles sizes were 60 nm, and 37 nm, respectively, as obtained by atomic force microscopy. Magnetic force microscopy imaging showed that the local magnetic structure changed from disordered stripe domains of about 200 nm for Ni and Ni(N) to a structure without a magnetic contrast, indicating the paramagnetic state of this material, which confirmed the structural transformation from Ni to Ni_3N.

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1. Introduction

In thin film growth processes, the properties of the film deposited by different physical vapor deposition methods are function of their composition, their structure and their microstructure. These three points are fixed by the deposition conditions: the deposition rate, the substrate temperature, the ion/atom bombardment, and the contents of impurities. By changing the main deposition parameters, the energy delivered to the growing film can be influenced, thus allowing the formation of nanocrystalline films with different grain-orientation and chemical composition [1–3]. The advantage of sputtering methods is that they enable the replacement of the substrate heating with particle energy (several eV compared with approximately 0.1 eV in the evaporation process), and thus the formation of films of high temperature phases. For instance, one variation of reactive sputtering in a nitrogen environment provides nitrogen atoms from backfilled molecular N_2 gas, and enables their incorporation into the growing film by sputtered and reflected energetic particles, modifying its physical properties at a relatively low substrate temperature [4–6].

The formation of metastable Ni_4N and Ni_3N phases with increasing nitrogen partial pressure during reactive dc sputtering of nickel in Ar/N_2 gas mixtures and N_2 plasma has already been reported [7,8]. However, by radio-frequency diode sputtering, the formation of the Ni_3N phase was observed at room temperature (RT) [9], while in a subsequent study involving the effects of substrate temperature, the formation of a Ni_3N phase was reported at both RT and 220 °C [10].

Magnetic characterization of nickel thin films showed that ferromagnetic Ni was transformed into a non-magnetic Ni_3N phase upon nitrogen implantation [11]. An interesting topic for relatively thick polycrystalline nickel films would be to establish the relation between the change in local structural and magnetic properties with nitrogen fraction. As atomic force microscopy (AFM) has become the most advantageous technique for imaging films structure on a nanometer scale, magnetic force microscopy (MFM) is becoming a promising technique for the assessment of local magnetic properties at high lateral resolution [12].

In this article, the influence of nitrogen partial pressure during the reactive ion sputter deposition of nickel and nickel nitride films on structural changes, were investigated using X-ray diffraction (XRD) technique, for the substrate at RT and at the temperature of 100 °C. The nitrogen and argon contents in the Ni(N) thin films were measured by Auger electron spectroscope (AES). The morphology of obtained thin films and the effect of chemical

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composition and structural changes on the local magnetic properties were studied by the AFM and MFM techniques. The aim of this work was to demonstrate the ability of MFM analysis to follow the changes of the magnetic properties of the thin films of nickel starting from ferromagnetic Ni and Ni(N) to its final transformation into a paramagnetic (non-magnetic) Ni_3N phase and to correlate AFM and MFM results to determine the structure of particular magnetic domains.

2. Experimental procedure

The Ni(N) films were deposited on glass substrates using a dc triode sputtering of a Ni (99.99% purity) target with Ar^+ ions in a Balzers Sputtron II vacuum system under an argon atmosphere or under a partial pressure of nitrogen of 0.013 Pa or 0.04 Pa. Thus the nitrogen content, defined as $p_{N2}/(p_{Ar} + p_{N2})$, was 0%, 9% or 23%. The residual pressure in the chamber was 7×10^{-4} Pa and the partial pressure of argon was 0.13 Pa. Presputtering was performed for 10 min with Ar^+ ions. The triode sputtering apparatus consists of two vacuum parts: the ionization chamber and the deposition chamber. The plasma is vertically confined in the central part of the deposition chamber and does not come in contact with the chamber walls and with the substrate holder. The geometry of the triode sputtering system enable the substrate holder to rotate around two axes at the distance of 300 mm from the central Ni target. The central target diameter was 60 mm. The acceleration voltage (U = 1.3 kV) and the current on the target (I = 0.7 A) were maintained constant during the experiment. The glass substrates (Corning 7059) were cleaned by the standard cleaning procedure prior to each nickel deposition. Two sets of samples for different N_2 gas content in the chamber were prepared: the first set was with the substrate at room temperature, and the second with a substrate temperature of 100 °C. The thickness of the deposited nickel films was about 600 nm as measured using a profilometer Talystep I.

The phase identification and preferred orientation of deposited films were performed by XRD analysis using a Siemens D500 diffractometer supplied with software Diffracplus with Ni-filtered Cu Kα radiation and a scanning speed of 0.02∘2θ/s. The phases were identified according to the JCPDS-ICDD-PDF data base [13].

The nitrogen and argon contents in the Ni(N) thin films, deposited at RT and at 100 °C, were measured by AES PHI SAM model 545A (PerkinElmer). Depth profile analysis was made using electron beam of energy 3 keV, a beam current of 1 mA and beam diameter of about 40 μm. The samples were ion sputtered with two inclined beams of 1 keV Ar^+ ions.

The sheet resistance, $R/\square$ of the films was measured by the four-point probe technique. The electrical resistivity values, ρ, were deduced from the sheet resistance and the film thickness, d, using the relation $R = \rho/d$.

Imaging of the topography and magnetic domains was performed with scanning probe microscope (SPM), Quadrex Multimode IIIe (Veeco Instruments Inc.), operated under ambient conditions. The topography was acquired in the main scan by standard tapping mode AFM using a commercial Veeco MESP tip. For the MFM measurements, the tip was subsequently retraced in the lift mode by a constant tip-sample distance of 40 nm to minimize the influence of the topography during the magnetic imaging. The tip was magnetized in the z-direction parallel to the tip axis. With a phase shift in the cantilever oscillation, the tip responds to changes in the magnetic force and the positive or negative shift results from a repulsive or attractive interaction in the out-of plane direction. The root mean square (rms) surface roughness and the rms of the phase shift were estimated using the subprogram packages of the SPM as the average values of images taken from different points within each sample surface.

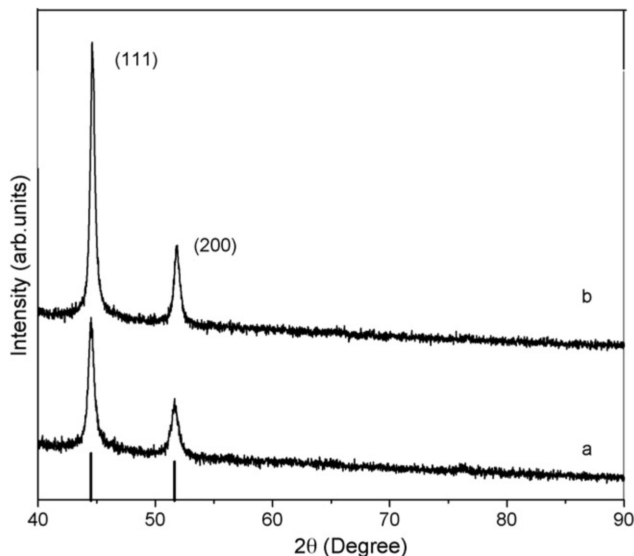

Fig. 1. XRD patterns of Ni films deposited with no N_2 in the sputtering chamber at: (a) RT and (b) 100 °C.

3. Results and discussion

The XRD patterns for Ni films deposited onto a glass substrate at RT and 100 °C under Ar sputtering with no N_2 in the vacuum chamber are shown in Fig. 1. The XRD data were obtained in the 2θ range from 40° to 90°. The peak intensities for Ni films are very different from those of Ni powder [13] and only the (1 1 1) and (2 0 0) peaks appear in the patterns. In addition, a substantial increase in the (1 1 1) peak height was observed for the films deposited at 100 °C (Fig. 1b) with a tendency to enhance the preferred (1 1 1) orientation. The d values and lattice parameters derived from the (1 1 1) and (2 0 0) peaks, given in Table 1, are nearly the same as the standard Ni values. According to the literature [14], an increase in the temperature should promote the growth of the crystallographic planes with the highest atom density. A perpendicular texture is commonly found in vapor deposited thin films on amorphous and polycrystalline substrates [15]. The origin of this alignment is the typical minimization of the surface and interface energies producing, as a consequence, the (1 1 1) orientation for a face centered cubic (fcc) lattice. Therefore, Ni films with a highly preferred (1 1 1) orientation were deposited by sputtering at 100 °C. Activation is enabled by the energy of the sputtered Ni atoms (several eV) and the higher substrate temperature. For e-beam evaporation in a triode ion plating system with argon ions bombardment of the growing film, the (1 1 1) preferred orientation increases with the energy delivered to the nickel film [16].

The XRD patterns for Ni films deposited under Ar sputtering at RT and 100 °C with a 9% N_2 are shown in Fig. 2. A substantial increase in the (2 0 0) peak height was observed for the higher

Table 1
Measured and calculated values of d_{hkl} and a for nickel films deposited with no N_2 and 9% N_2 gas fraction.

N_2 (%)	T_{sub} (°C)	$d_{(1\,1\,1)}$ (nm)	$d_{(2\,0\,0)}$ (nm)	a (nm)
0	RT	0.2036	0.1772	0.3534
	100	0.2032	0.1766	0.3523
9	RT	0.2057	0.1811	0.3592
	100	0.2036	0.1789	0.3557
Ni st 4-085		0.2034	0.1762	0.3524

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