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The influence of thickness on magnetic properties of nanostructured nickel thin films obtained by GLAD technique



J. Potočnik^a, M. Nenadović^a, N. Bundaleski^a, B. Jokić^b, M. Mitrić^a, M. Popović^a, Z. Rakočević^{a,*}

^a University of Belgrade, INS VINČA, Mike Petrovića Alasa 12-14, 11001 Belgrade, Serbia
^b University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

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1. Introduction

Nickel is a ferromagnetic transition metal that has found wide application in the form of nanostructured thin films. It can be used in different fields such as ferrofluid technology, magnetic resonance imaging [1-3], as solar absorbers for photo thermal conversion [4], electrochemistry [5], nanotechnology, microelectronic devices [6], etc. In particular, ferromagnetic thin films are essential constituents of multilayer systems, representing new magnetic sensors based on giant magnetoresistance and similar effects (e.g. tunneling magnetoresistance or spin valve magnetic giant magnetoresistance). These devices are nowadays used to read data in hard disk drives, biosystems and microelectromechanical systems, which is the main driving force for studying the magnetic properties of ferromagnetic thin films, including those of nickel. It is well-known that nickel magnetization originates from the spin polarization of the 3d electrons, the latter being sensitive to the local environment i.e. to the thin film morphology, structure and composition [7]. The physical (electrical, structural, optical and magnetic) and chemical (amount of impurities) properties of nickel thin films strongly depend on the

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ABSTRACT

In this work, nickel (Ni) thin films were deposited by electron beam evaporation of Ni using Glancing Angle Deposition technique onto the glass substrate with the thickness varied from 25 nm to 150 nm. Characterization of obtained Ni films was performed by scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and by Magneto-Optical Kerr effect measurements (MOKE). The effect of thickness on structural, chemical and magnetic properties of nickel films has been studied. Observed changes in microstructure were correlated with the variation in magnetic parameters obtained by MOKE measurements. It was found that for thinner Ni films, the enhancement of coercivity is due to the surface roughness of Ni films, while for thicker films the observed asymmetry of coercivity is due to the mechanism of column size growth. © 2016 Elsevier Ltd. All rights reserved.

deposition technique, deposition parameters and its thickness [8– 10]. Hemmous et al. examined the effect of several substrates and of thickness in a nanometer range on the magnetic properties of thermal evaporated Ni thin films [11]. They have noted a monotonous increase of the coercive field and saturation with thickness as well as the transition of the magnetization easy axis from in-plane to out-of-plane, which is associated with a stressinduced anisotropy. Results of studies of some structural and magnetic properties of thin Ni films deposited on different substrates using a thermal evaporation were presented in many reports [12–14].

Glancing Angle Deposition (GLAD) technique is a physical vapor deposition process where the incoming vapor atoms arrive on the substrate from an oblique angle of incidence, creating different film structures such as slanted and vertical posts [15,16], zig-zag [17] or helices [18]. The main property of this method is atomic shadowing mechanism, leading to preferential growth of existing nuclei and islands at the substrate surface. As these features grow, the shadowing effect is enhanced and promotes the formation of columnar structures [19]. Nickel thin films deposited at oblique incidence exhibit a magnetic anisotropy with an easy axis parallel to the incidence plane [20]. T. Otiti [21] found that in the film plane, the direction of easy magnetization lies parallel to the incidence plane at high incidence angles and the column becomes needlelike. The obtained results indicate that there is the extreme

Corresponding author.
 E-mail address: zlatkora@vinca.rs (Z. Rakočević).

sensitivity of the magnetic behavior of obliquely deposited films to the microstructural morphology. As already stated, the thickness of nickel thin films strongly influence their magnetic properties. However, to the best of our knowledge, there are no detailed studies of thickness-dependent magnetic properties of nanostructured Ni thin films obtained by GLAD.

In this paper, we have deposited nickel thin films thicknesses in the range 25–150 nm on glass substrate using GLAD technique. The detailed structural, chemical and magnetic analysis of Ni films was performed. The variation of magnetic properties of deposited samples can be directly correlated to the observed changes in structural parameters. The obtained results reveal the possibility to tailor the magnetic properties of nanostructured nickel thin films deposited by GLAD only by controlling the thin film thickness.

2. Experimental procedure

Nickel thin films were deposited by electron beam evaporation of nickel (99.98% purity, Onix-Met) using Glancing Angle Deposition technique by programming the vapor incidence angle (substrate tilt) and substrate azimuthal rotation. The substrates used were microscope slide glass plates. Prior to deposition, glass substrates were cut into 1×0.5 cm² pieces, cleaned with ethanol and rinsed with 18.2 M Ω deionized water in ultrasonic bath. After that, they were additionally cleaned by exposing to ozone for 20 min (NovaScan PSD-UVT ozonizer) and then attached to substrate holder in the chamber. Glass substrates were located about 20 cm away from the nickel target, and oriented in such way that the angle between the surface normal and the direction of the flux of evaporated nickel atoms is 65°. Base pressure in the chamber was 4×10^{-5} Pa. The substrates were rotated with a suitable constant speed (\sim 40 rpm) during the deposition, so that the columns grew parallel to the surface normal. Nickel thin films with thicknesses in the range from 25 to 150 nm were deposited at a rate of ~2.5 nm/min. Thickness of the films was controlled in-situ by quartz crystal thickness monitor and measured using crosssectional SEM micrographs.

Field Emission Scanning Electron Microscope, Mira XMU TESCAN was used for morphology studies. The samples were analyzed in cross-sectional view and the acceleration voltage between cathode and anode was equal to 20 kV. The surface topography of the films was analyzed using Atomic Force Microscopy Multimode Quadrex SPM with Nanoscope IIIe controller (Veeco Instruments, Inc.). AFM was operated in the tapping mode, using a commercial Veeco RFESP probe (Phosphorus (n) doped Si) with a cantilever length of 225 μ m. The structure of nickel films was examined by XRD analysis using a Philips PW1050 with CuK α emission with λ = 0.15418 nm. The samples were recorded in a 2 θ range of 35°–55°. X-ray Photoelectron Spectroscopy analysis of samples was carried out using SPECS System with

XP50M X-ray source and PHOIBOS 100/150 analyzer. The measurements were performed in a low 10⁻⁶ Pa range using a monochromatic Al K α X-ray source (photon energy of 1486.74 eV) and eflood gun was used to compensate the electron emission and prevent sample charging. In order to remove the surface impurities, the samples were sputtered with 5 keV argon ions until steady surface composition was reached. Magneto-Optical Kerr effect Microscope (Evico Magnetics GmbH) was used for determining the magnetic properties of deposited thin films. Magnetic hysteresis loops were recorded in the longitudinal (LMOKE) and polar (PMOKE) mode of operation. The measurements were performed at room temperature in the range of the magnetic field from -500 Oe to 500 Oe. For each sample, LMOKE loops were measured as a function of in-plane azimuthal angle (ϕ) from 0° to 360° with 20° increment.

3. Results and discussion

The cross-sectional structures of different thickness Ni films were studied by SEM and the micrographs of corresponding samples are given in Fig. 1. The measured thickness of the films is in the range of 25–150 nm. In all micrographs the presence of small particles of Au-Pd alloy deposited over the whole sample (i.e. over the non-conductive glass substrate and the nickel film) can be observed, which is necessary for obtaining SEM imaging. Nevertheless, it is clearly visible that the growth of nickel thin films under the given deposition conditions led to the formation of well defined columns perpendicularly oriented with respect to the surface. The column width is changing during the growth. Initially they are guite narrow whilst, after the initial nucleation is completed, their width gradually increases with the length. One can also notice that some of the columns stopped growing, thus enabling the surviving columns to grow further in both, height and diameter. The direct consequence of the column widening with the growth is the gradual increase of the column width with the thin film thickness. The column diameter at the film surface changes from 12 nm, for the smallest film thickness of 25 nm, till 34 nm in the case of the 150 nm thick film. At the same time, the surface roughness appears to increase with the surface thickness. The increase of the column width with the thin film thickness is the well-known feature of nanostructured thin films deposited by GLAD, which is caused by the shadowing effect. Indeed, while different kinds of non-uniformity is observed in many GLAD structures, the most investigated one is column broadening in the vertical column morphology caused by competitive growth and the column-to-column structural variation associated with random film growth effects. With increasing film thickness, the nanocolumnar features bundle together to form the larger structure. Consequently, the thin film morphology consists of



Fig. 1. Cross-sectional SEM images for: (a) 25 nm, (b) 50 nm, (c) 80 nm, (d) 120 nm and (e) 150 nm Ni thin films.

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