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Structural, Chemical and Magnetic Properties of Nickel Vertical Posts Obtained by Glancing Angle Deposition Technique

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Abstract:

In this work, Glancing Angle Deposition technique was used for obtaining nanostructured nickel thin film with vertical posts on glass substrate which was positioned 75 degrees with respect to the substrate normal and rotated with a suitable constant speed. The obtained nickel thin film was characterized by Scanning Electron Microscopy, Atomic Force Microscopy and X-ray Photoelectron Spectroscopy. It was found that the deposited thin film consists of 94.0 at.% of nickel. Magnetic properties of the deposited thin film were determined by Magneto-Optical Kerr Effect Microscopy. According to the obtained coercivity values, it can be concluded that the nickel thin film shows uniaxial magnetic anisotropy.

Keywords: *Glancing Angle Deposition, XPS, AFM, Magnetic properties, Nickel thin film.*

1. Introduction

Nickel is a metallic magnetic material that has found wide application in the form of thin films. It can be used in solar thermal energy conversion [1], ferrofluid technology, magnetic resonance imaging [2-4], high density recording media [5], electrochemistry, nanotechnology, microelectronic devices, solar energetic [6], etc. Nickel nanowires have application in sensors [7,8] as well as catalyst for the growth of carbon nanotubes [9]. Also, nickel has been widely used as a deposit material for fabrication of multilayer monochromators for neutron optics [10]. Desired morphology of nickel thin films can be achieved by selecting an appropriate substrate and the deposition technique [11-13].

Glancing Angle Deposition (GLAD) is a physical vapour deposition technique for obtaining columnar nanostructures. This technique is based on an accurate moving of the substrate during the deposition as illustrated in Fig. 1. In this method, the substrate can be rotated around two axes. Rotation around one axis is changing the angle of the incident vapour flux (tilt angle) while the rotating about the other one control the structure of the deposited film (azimuthal angle) [15]. If the tilt angle is kept fixed during the deposition, the structure of the deposited thin film consists of nanocolumns inclined towards the direction of the incident vapour flux. When uniform azimuthal rotation is applied to the substrate, the resulting nanostructures are composed of vertical columns normal to the substrate surface [16].

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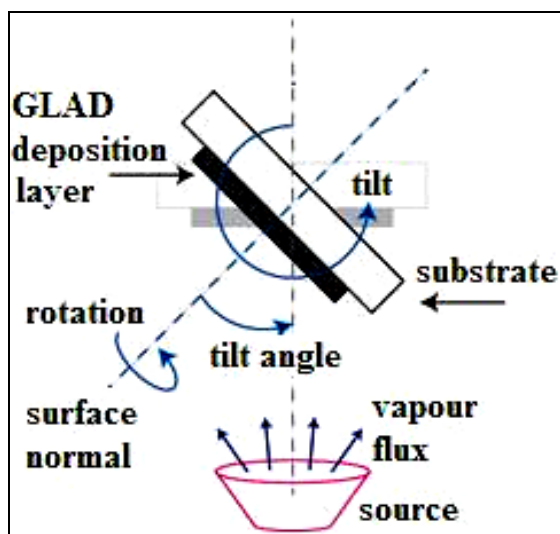


Fig. 1. Schematic view of GLAD apparatus [14].

The aim of this study was to demonstrate the utility of the GLAD growth technique for fabricating thin films composed of vertical columns. The obtained thin film was characterized by Scanning Electron Microscopy, Atomic Force Microscopy, X-ray Photoelectron Spectroscopy and Magneto-Optical Kerr Effect Microscopy.

2. Experimental procedure

Nickel thin film was deposited by evaporating nickel onto a Thermo Scientific microscope slide glass. Prior to deposition, the glass substrate was cut by a Low Speed Diamond Wheel Saw (Southbay Technology, Inc.), cleaned in ethanol solution in ultra-sonic bath and rinsed in 18.2 M Ω deionized water. Glass substrate was positioned at the tilt angle of 75 degrees with respect to the substrate normal and rotated along its azimuth by a suitable constant speed. The base pressure of the system was 4×10^{-5} Pa. Emission current was kept constant to the value of 170 mA. The time of deposition was 1 hour and 30 minutes.

Field Emission Scanning Electron Microscope, Mira XMU (TESCAN, Czech Republic) at 20 kV was used for morphology studies. Prior to the FESEM analysis, the sample was prepared cross-sectionally and sputter coated with Au-Pd alloy as a conducting material for SEM imaging. Cross sectional SEM image of the sample was used to determine the thickness of the film.

The obtained nickel thin film was also characterized using Multimode Quadrex SPM with Nanoscope IIIe controller (Veeco Instruments, Inc.). In this work, Atomic Force Microscope (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 measurement was made in air, at room temperature.

X-ray Photoelectron Spectroscopy (SPECS Systems, PHOIBOS 100/150) was used to determine the composition of the deposited thin film. 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). Detailed spectra were taken in FAT20 mode with energy step of 0.1 eV and dwell time of 1–2 s depending on the considered line. To remove the surface impurities the sample was sputtered for 20 minutes using 5 keV argon ions. The surface composition of thin film was determined from the high-resolution spectra of Ni2p, O1s and C1s lines.

Concentrations of elements present in the film were obtained using CasaXPS software.

Magneto-Optical Kerr effect Microscope (Evico Magnetics GmbH) was used for determining the magnetic properties of the deposited thin film. This effect describes the interaction of electromagnetic plane polarized waves with the analyzed magnetic material. More precisely, this technique measures the angle of rotation of the polarization plane due to the light reflection from the magnetic material placed in magnetic field [17]. Magnetic hysteresis loops were recorded at the longitudinal mode of operation, which means that the magnetization is in the plane of the sample. Measurements were performed at the room temperature in the magnetic field range from -1500 Oe to 1500 Oe.

3. Results and Discussion

The structure of nickel thin film was studied by SEM and the cross-sectional morphology of the film is given in Fig. 2.

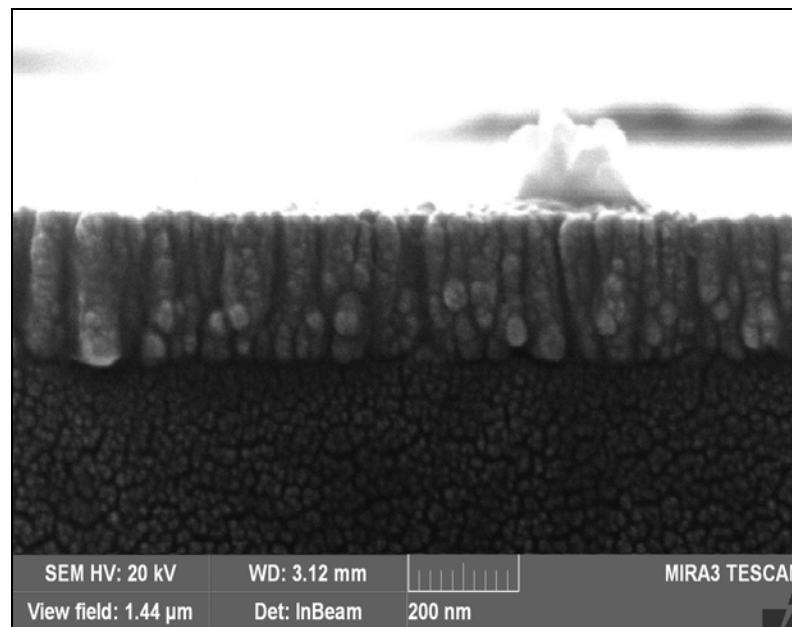


Fig. 2. Cross-sectional SEM image of the deposited nickel thin film.

It can be seen that the thin film consists of vertical posts and the diameter of the columns is (34 ± 5) nm. The thickness of the deposited nickel thin film is (270 ± 10) nm. Further, surface morphology of nickel thin film was analyzed by AFM and typical topography AFM image of as prepared nickel thin film is presented in Fig. 3.

Based on AFM image, it was found that the diameter of the columns is (35 ± 5) nm, which shows good agreement with the values obtained by SEM analysis. The surface roughness of nickel thin film, which was defined as the square deviation of an analyzed surface from the ideal, was estimated from AFM image and it was found to be (2.1 ± 0.1) nm.

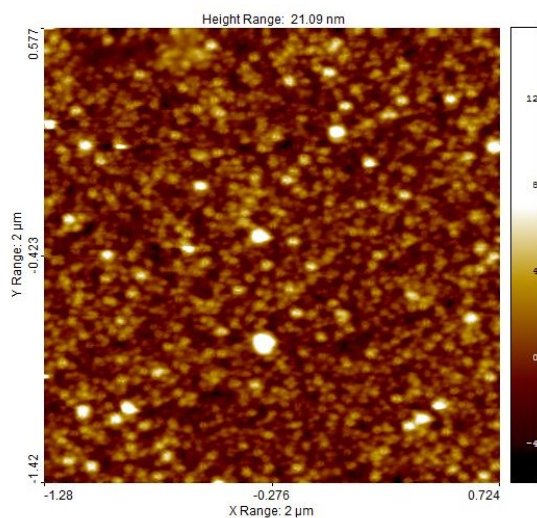


Fig. 3. Topographic AFM image of the nickel thin film.

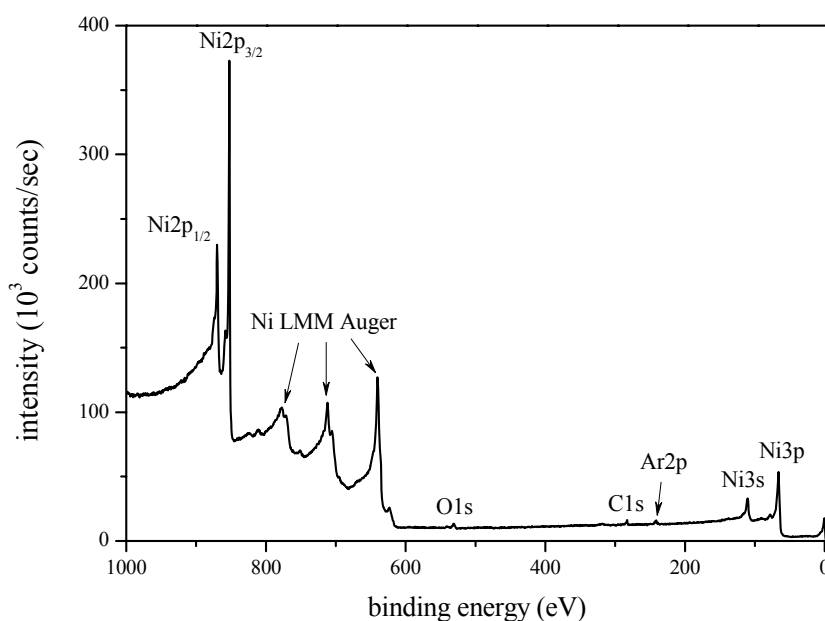


Fig. 4. Survey XPS spectra of the deposited thin film.

Survey XPS spectrum taken after the surface cleaning by the ion beam is shown in Fig. 4. It contains the characteristic lines originating from Ni (Ni2p, Ni3s and Ni3p), O (O1s) and C (C1s) as well as Auger transition lines from Ni. Small amounts of carbon, which is a typical contaminant, were detected at the Ni thin film surface. The Ar2p peak identified in the spectrum comes from the implanted argon during cleaning the surface. The Ni atomic concentration was calculated using the area of Ni2p_{3/2} region only. It was found that the thin film consists of 94.0 at.% of nickel, 2.9 at.% of oxygen and 3.1 at.% of carbon, which is similar to the values obtained for bulk nickel (95.3 at.% of nickel, 1.6 at.% of oxygen and 3.1 at.% of carbon). The detailed spectra of the Ni2p_{3/2} and O1s core levels are shown in Fig. 5. Ni2p_{3/2} line (Fig. 5.a) is clearly asymmetric, being typical for metallic samples. Therefore, this line was modeled as a superposition of 3 peaks, centered at 852.6 eV, 856.2 eV and 858.6 eV, with GL30 pseudo-Voigt profiles, by applying constraints (relative positions, peak widths and relative intensities) for metallic nickel [18]. Detailed spectrum recorded from 537 eV to 525

eV for O1s is shown on Fig. 5.b. It can be seen that the O1s peak is relatively broad and asymmetric as it is associated with different types of bonds. Further deconvolution revealed four distinct components, the strongest peak locating at 531.1 eV originating from Ni(OH)₂ while the peak at 529.8 eV belongs to NiO bond [18]. Other two peaks at 532.5 eV and 534.3 eV are associated with C-O-C and H-O-H, respectively [19].

Measuring the longitudinal MOKE at varying azimuthal angles (φ) the in-plane easy axis and hard axis were determined. To vary the azimuthal angle, the sample was rotated in the magnetic field. Azimuthal dependence of the coercivity values for as prepared nickel thin film was presented in Fig. 6.

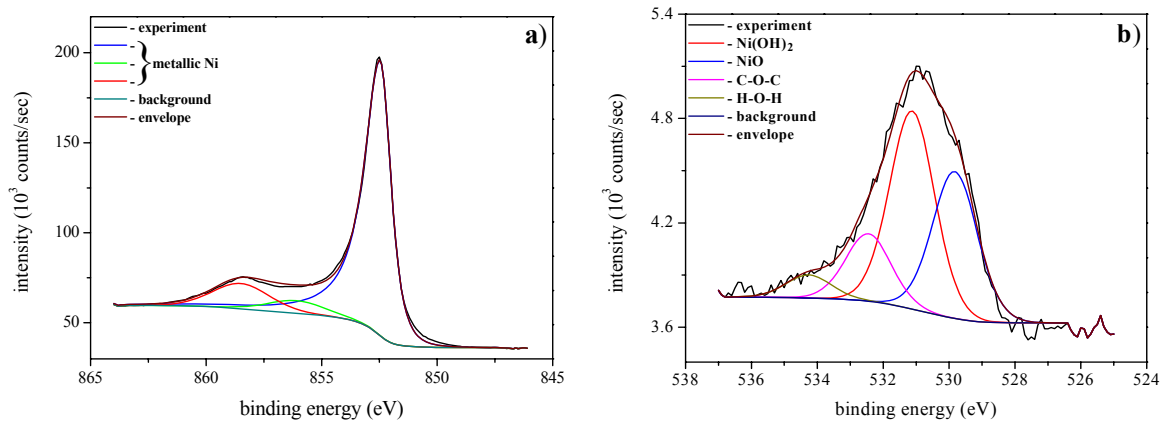


Fig. 5. High-resolution spectra of the a) Ni2p_{3/2} and b) O1s regions of the deposited thin film.

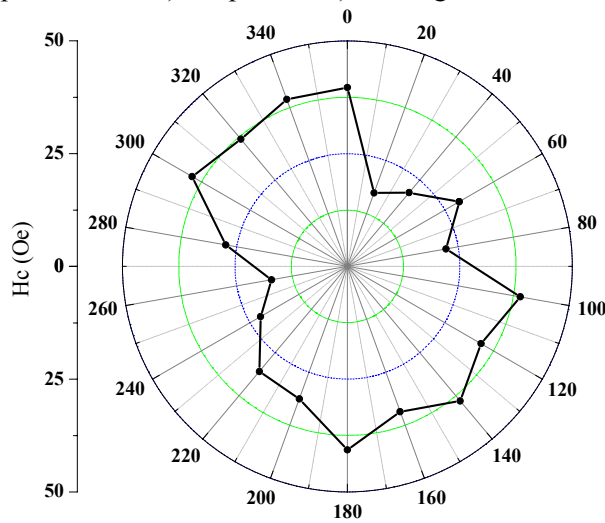


Fig. 6. Coercivity values of the nickel thin film as a function of the sample rotation.

It was observed that the diagram has an asymmetrical shape, which means that the coercivity values were changed depending on the angle of the sample rotation. The value of coercivity is the smallest at $\varphi = 260$ degrees and it is equal to $H_c = (17 \pm 2)$ Oe. It can be concluded that the axis along $\varphi = 260$ degrees represents the hard axis of magnetization in the thin film plane. Comparing with other coercivity values, coercivity at $\varphi = 180$ degrees has the highest value, $H_c = (41 \pm 5)$ Oe, and this is evidence of the easy axis of magnetization. The coercivity value at $\varphi = 0$ degrees is similar to that of $\varphi = 180$ degrees and the obtained coercivity is equal to $H_c = (40 \pm 5)$ Oe. According to obtained results it can be concluded that the nanostructured nickel thin film has in-plane uniaxial magnetic anisotropy.

4. Conclusion

In this paper, we have demonstrated that the deposition of nickel thin film with vertical posts can be achieved using GLAD technique. The obtained nickel thin film, thickness of 270 nm, has a columnar structure with the diameter of the columns of 35 nm. According to AFM analysis it was found that the surface roughness of the deposited thin film is 2.1 nm. As confirmed by XPS analysis, the obtained thin film consists of 94.0 at.% of nickel. The values of the coercivity, obtained from the magnetic hysteresis loops, indicated that the nickel thin film has a uniaxial magnetic anisotropy.

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Садржај: У овом раду метода депоновања при малим угловима користила се за добијање наноструктурног танког слоја никла који се састоји од вертикалних стубића. Узорак је депонован на подлогу од стакла, која се, у односу на нормалу на подлогу, налазила под углом од 75 степени. Током депоновања, подлога од стакла је ротирана константном брзином. Добијени танки слој никла анализиран је помоћу сканирајуће електронске микроскопије, микроскопије у пољу атомских сила и рендгенске фотоелектронске спектроскопије. Нађено је да је удео никла у депонованом узорку 94 ат.%. Магнетна својства танког слоја никла посматрана су микроскопом који је заснован на магнетно-оптичком Керовом ефекту. На основу добијених резултата може се закључити да танки слој никла посједује магнетну анизотропију у правцу једне осе.

Кључне речи: депоновање при малим угловима; рендгенска фотоелектронска спектроскопија; микроскоп у пољу атомских сила; магнетна својства; танки слој никла.

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