Science of Sintering, **45** (2013) 61-67

doi: 10.2298/SOS1301061P

UDK 532.74

Structural Characterization of the Nickel Thin Film Deposited by GLAD Technique

J. Potočnik[1], M. Nenadović[1], B. Jokić[2], S. Štrbac[3], Z. Rakočević[1*)]
[1]INS Vinča, Laboratory of Atomic Physics, University of Belgrade, Mike Alasa 12-14, 11001 Belgrade, Serbia
[2]Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia
[3]ICTM-Institute of Electrochemistry, University of Belgrade, Njegoševa 12, 11000 Belgrade, Serbia

Abstract:
In this work, a columnar structure of nickel thin film has been obtained using an advanced deposition technique known as Glancing Angle Deposition. Nickel thin film was deposited on glass sample at the constant emission current of 100 mA. Glass sample was positioned 15 degrees with respect to the nickel vapor flux. The obtained nickel thin film was characterized by Force Modulation Atomic Force Microscopy and by Scanning Electron Microscopy. Analysis indicated that the formation of the columnar structure occurred at the film thickness of 1 μm, which was achieved for the deposition time of 3 hours.

Keywords: *Glancing Angle Deposition, Force Modulation Atomic Force Microscopy, nickel, cross section*

1. Introduction

Nickel is a material that has found wide application in the form of thin films and that is essential for modern technologies based on the fabrication of nanostructured materials. Desired morphology of nickel thin films can be achieved by selecting an appropriate substrate and the deposition technique [1-3]. Chemical, optical, electrical, magnetic and other properties of nickel thin films significantly differ from the same properties of bulk materials, and can be determined with high degree of accuracy by the use of modern techniques [4-6]. Glancing Angle Deposition (GLAD) technique appeared to be very promising for thin films deposition [7], and since a hand made instrument is used in this work, this technique will be briefly described.

GLAD is a very flexible technique for the deposition of columnar nanostructures, which is based on an accurate moving of the substrate during the deposition as illustrated in Fig. 1 by a schematic diagram [12]. Obtained columnar nanostructures are inclined toward the direction of the incident vapor flux and may take a form of slanted and vertical posts [8,9], helices [10], or zig - zag [11] structures, as well as their combination. GLAD technique is compatible with a variety of materials, which increases its combinatorial power [13].

Schematic illustration of the growth of columnar nanostructures is given in Fig. 2. Incident vapor flux is represented by vector F (Fig. 2a). The formation of the nucleus from the

---

*) **Corresponding author**: zlatkora@vinca.rs

arriving vapor flux is a random process (Fig. 2b). Nuclei grow into columns giving rise to the development of the shadowing (Fig. 2c). The columns are not equal in size and as a result, some columns will screen their neighbors from the vapor flux causing the decrease of their growth. At some point, large columns entirely overshadow small grains and columns whose further growth stops. As columns grow, the more the incident vapor flux deposits on them. This self – reinforcing behavior leads to the development of isolated columns. The lateral component represents the source for shadowing effect, while the vertical component remains constant during deposition.

The column tilt angle (β) is smaller than the incident vapor flux angle (α) (Fig. 2d). Its value can be obtained using the tangent rule:

$$\tan\beta = \frac{1}{2}\tan\alpha$$

for small α values [14,15]. The size and density on the nanostructures will change as function of the incident vapor flux angle (α).

Films obtained by GLAD technique are robust, so other methods can be used to modify the structure, such as annealing [16-22], oxidation [23,24] and etching [25-27]. Many of these techniques may be applied in parallel, producing a better structure quality. The structure of thin films can be adjusted to suit the needs of the applications. With the development of the GLAD technique, researchers were able to manipulate the columnar structure by shifting the position of the substrate during the deposition [13].

In this work, GLAD instrument designed in our laboratory was used for the deposition of nickel thin films with columnar structure. Surface morphology and cross sectional images of obtained samples were acquired by Atomic Force Microscopy (AFM) and by Field Emission Scanning Electron Microscopy (FESEM).

2. Experimental

Nickel thin film was deposited using GLAD technique by evaporating nickel onto glass substrate. Deposition of nickel was performed in the vacuum chamber. The base pressure of the system was 6.7 x 10^{-5} Pa. Emission current was constant and its value was 100 mA.

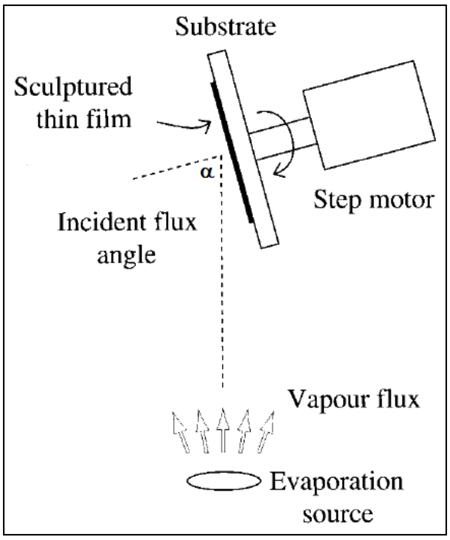

Fig. 1. Schematic diagram of the Glancing Angle Deposition technique; α represents the incident flux angle [12].

Glass substrate was cleaned in ethanol solution in ultra – sonic bath and rinsed with 18.2 MΩ deionised water. After that, substrate was also cleaned by the ozone (NovaScan PSD-UVT) and then attached to substrate holder in the chamber. Glass sample was positioned at the angle of 15 degrees with respect to the nickel vapor flux (angle αin Fig. 2a). The duration of the deposition was 3 hours.

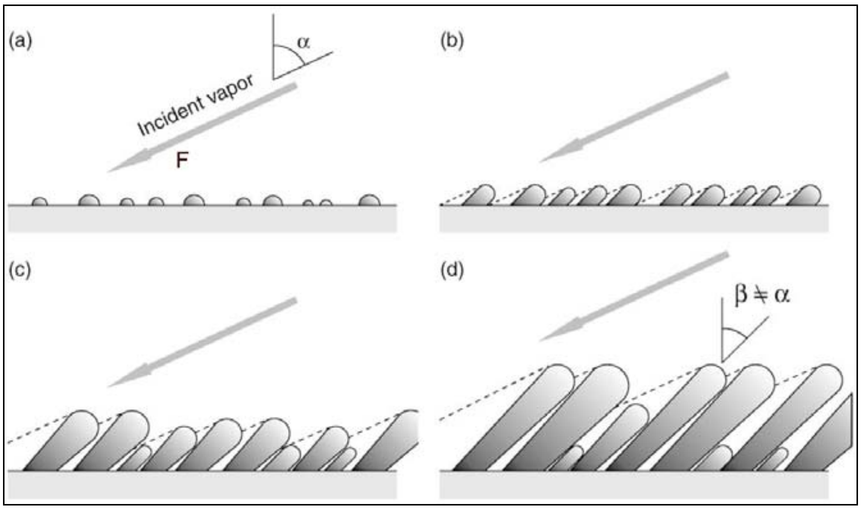

Fig. 2. Schematic view of GLAD growth: (a) initial arrival of vapor flux at an angle α; (b) nuclei grow; (c) columns develop; (d) columns grow at an inclined angle [13].

The obtained nickel thin films were characterized using Multimode Quadrex SPM with Nanoscope IIIe controller (Veeco Instruments, Inc.). Force Modulation AFM, further in the text reffered to as Force Modulation Microscopy (FMM) was used to acquire cross sectional images. In this mode, the elasticity of the sample is measured by cantilever oscillations in order to produce a weak indentation into the sample surface [28]. When the tip reaches the edge of the sample its oscillations stop, and when the tip goes back to the sample, the oscillations start again. This is the main advantage of FMM over standard AFM technique, which enables cross sectional imaging of the sample. Cross sectional FMM image of the sample was used to determine the thickness of the film.

Field Emission Scanning Electron Microscope, Mira XMU (TESCAN, Czech Republic) at 20 kV was used for additional morphology studies. Prior to the FESEM analysis, the sample was sputter coated with Au - Pd alloy. Compared to SEM, FESEM provided narrower probing beams at both low and high electron energies, resulting in improved spatial resolution and minimized sample charging and damage.

3. Results and discussion

The depth analysis of the structure of nickel thin film deposited on glass substrate by GLAD was performed by both FMM and FESEM techniques through cross section imaging of the sample. Fig. 3 shows three - dimensional (3D) FMM cross section image (3.5 x 3.5) μm^2 of the deposited nickel thin film, where there are three distinguished areas: dark brown area is air, a rough light brown area on the left side is glass substrate and an area between the glass substrate and the air is deposited nickel film. It is clearly visible that the growth of nickel thin film under given deposition conditions led to the formation of a columnar structure. These columns are not perpendicular to the surface of the substrate, but tilted at a certain angle.

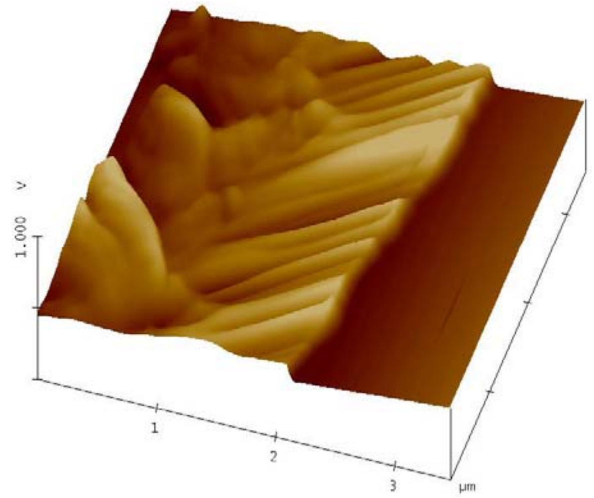

Fig. 3. Cross sectional 3D FMM image (3.5 x 3.5) μm^2 of the nickel thin film.

Top view FMM cross section image of the same sample region enabled the accurate depth analysis and the estimation of the angle at which nickel columns are tilted with respect to the substrate surface as illustrated in Fig. 4. It is estimated that the columns are tilted at the angle of $\beta = (57 \pm 4)$ degrees, which is in a good agreement with the theoretically obtained value of $\beta = 60$ degrees, calculated for the deposition parameters used.

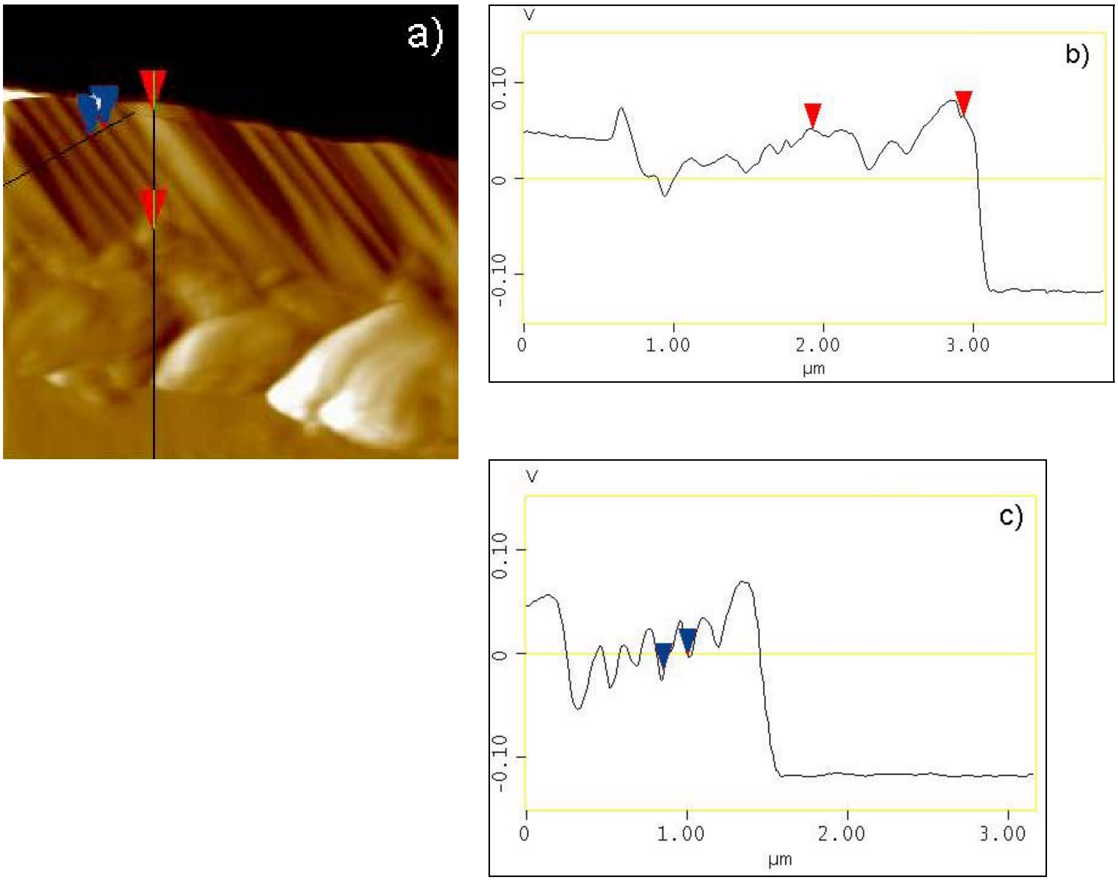

Fig. 4. FMM image analysis of the nickel thin film: (a) cross section image (4.0 x 4.0) μm^2; (b) determination of the sample thickness and (c) of the column diameter.

Depth analysis of the nickel thin film, was performed along lines drawn in Fig. 4a. Both, the estimation of the film thickness and the determination of the columns diameters were performed along designated lines between red and blue markers, respectively. Analysis presented in Fig. 4b indicates that the formation of the columnar structure occurs at the film thickness of (1.1 ± 0.2) μm. Diameter of the column, which was estimated from Fig. 4c, as the distance between blue markers, was (150 ± 50) nm.

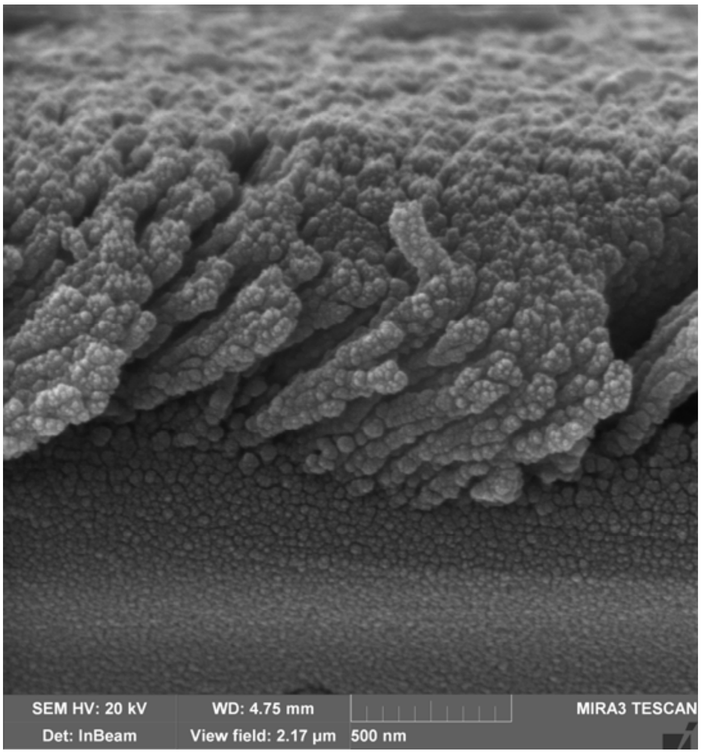

Fig. 5. FESEM cross section image of the nickel thin film deposited on glass substrate.

In order to support and confirm the results obtained by FMM, the cross sectional imaging of the sample was also performed using FESEM technique. From the image presented in Fig. 5, one can see that starting from the substrate surface, nickel columns are narrower in diameter and that after the initial nucleation is completed, they grow further achieving approximately constant diameter. The columns are densely packed and uniformly tilted across the substrate surface with almost the same length. One can also notice that the columns as well as the substrate are not flat like in FMM cross section image of the sample. The presence of small particles over the whole sample originates from the coating of the sample with Au - Pd alloy, which was necessary as a conducting material for SEM imaging since glass substrate is an insulator.

4. Conclusion

In this work, we have demonstrated that the deposition of nickel thin film with a columnar structure can be achieved using GLAD technique. Both Force Modulation Microscopy and Field Emission Scanning Electron Microscopy techniques proved to be suitable for the characterization of obtained columnar structures through cross sectional imaging of the nickel thin film sample. The image analyses have shown that the thickness of the nickel thin film was 1 μm and that nickel columns 150 nm in diameter are tilted at the angle of approximately 57 degrees with respect to the substrate surface.

Acknowledgement

This work was financially supported by the Ministry of Education and Science of the Republic of Serbia; project no. III45005.

5. References

1. N. Popović, Ž. Bogdanov, B. Goncić, S. Zec, Z. Rakočević, Thin Solid Films 343 (1999) 75
2. Z. Rakočević, S. Štrbac, R.J. Behm, Thin Solid Films 517 (2008) 709
3. M. Spasojević, A. Maričić, L. Rafailović, Science of Sintering 36 (2004) 105
4. Z. Rakočević, N. Popović, Z. Bogdanov, B. Goncić, S. Štrbac, Rev. Sci. Instrum. 79 (2008) 066101
5. N. Popović, Ž. Bogdanov, B. Goncić, S. Štrbac, Z. Rakočević, App. Surf. Sci. 255 (2009) 4027
6. M. Nenadović, S. Štrbac, Z. Rakočević, App. Surf. Sci. 256 (2010) 1652
7. T. Otiti, G. I. Ekosse, S. T. Stephen, J. Appl. Sci. Environ. Manage 11 (2007) 57
8. R. Messier, T. Gehrke, C. Frankel, V. C. Venugopal, W. Otano, A. Lakhtakia, J. Vac. Sci. Technol. A15 (1997) 2148
9. K. Robbie, M. J. Brett, J. Vac. Sci. Technol. A 15 (1997) 1460
10. Y. P. Zhao, D. X. Ye, G. C. Wang, T. M. Lu, NanoLett. 2 (2002) 351
11. R. Messier, V. C. Venugopal, P. D. Sunal, J. Vac. Sci. Technol. A 18 (2000) 1528
12. B. Dick, M. J. Brett, T. J. Smy, M. R. Freeman, M. Malac, R. F. Egerton, J. Vac. Sci. Technol. A 18 (2000) 1838
13. M. T. Taschuk, M. M. Hawkeye, M. J. Brett in Handbook of Deposition Technologies for Films and Coatings – Science, Applications and Technology, Third Edition (2010) 621
14. A. Lisfi, J. C. Lodder, Phys. Rev. B 63 (2001) 174441
15. J. M. Nieuwenhuizen, H. B. Haanstra. Philips Tech. Rev. 27 (1966) 87
16. Y. Sun, J. Gospodyn, P. Kursa et al., Appl. Surf. Sci. 248 (2005) 392
17. W. Smith, Z. Zhang, Y. Zhao, J. Vac. Sci. Technol. B25 (2007) 1875
18. P. C. P. Hrudey, M. Taschuk, Y.Y. Tsui et al., J. Vac. Sci. Technol. A23 (2005) 856
19. J. Gospodyn, M. Taschuk, P. Hrudey et al., Appl. Opt. 47 (2008) 2798
20. J. Kim, Z. Dohn´alek, B. Kay, Surf. Sci. 586 (2005) 137
21. E. Schubert, J. Fahlteich, Th. Hoche, G. Wagner, B. Rauschenbach, Nuc. Instr. Meth. Phys. Res. B244 (2006) 40
22. D. Flaherty, Z. Dohn´alek, A. Dohn´alkov´a et al., J. Phys. Chem. C111 (2007) 4765
23. X. Zhou, S. Virasawmy, W. Knoll et al., Plasmonics 2 (2007) 217
24. A. Lakhtakia, M. Horn, Optik 12 (2003) 556
25. S. Pursel, M. Horn, A. Lakhtakia, Opt. Eng. 46 (2007) 040507
26. K. Robbie, Y. Cui, C. Elliott et al., Appl. Opt. 45 (2006) 8298
27. M. Summers, M. Brett, Microelectron. Eng. 85 (2008) 1222
28. P. Maivald, H. J. Butt, S. A. C. Gould, C. B. Prater, B. Draine, J. A. Gurley, V. B. Elings, P. K. Hansma, Nanotechnology 2 (1991) 103

Садржај: *У овом раду, стубичаста структура танког слоја никла је добијена коришћењем напредне технике депоновања при малим угловима. Танки слој никла је депонован на стаклу, при константној емисионој струји која је износила 100 mA. Подлога од стакла је постављена, у односу на флукс паре никла, под углом од 15 степени. Добијени танки слој никла је карактерисан помоћу микроскопа у пољу атомских сила и сканирајућег електронског микроскопа. Анализа указује да се формирање стубичасте структуре јавља при дебљини слоја никла од 1 μm, а та дебљина је добијена након 3 сата депоновања.*

Кључне речи: *депоновање при малим угловима, микроскоп у пољу атомских сила, никл, попречни пресек*