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Understanding Nickel Thin Film crystallization using X-Ray Diffractometry

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ABSTRACT : Normal, helical and zigzag deposited Ni films were produced by letting a vapour stream of source material impinge on Corning 1737 glass substrates at oblique incidence while rotating the substrate during deposition. Films produced by glancing angle deposition (GLAD) technique while rotating the substrate. The microstructures of these Ni films were studied using X-ray diffractometry technique. The X-ray diffraction (XRD) patterns depicted 100% and 42% relative intensity (RI) peaks identified for normal and helical deposited Ni films but none for the zigzag deposited Ni film. Higher degree of crystallinity of Ni was demonstrated by the helical thin film sample having 200 nm thickness (sample Ni40) compared to the normal thin film which had only the 100% RI peak defined. Should an application therefore require Ni thin films of high crystallinity, it would be the film prepared with helical microstructure of 200 nm thickness that will be employed. @JASEM

Geologically, nickel occurs either as residual concentrations of nickeliferous laterites associated with basic/ultra basic igneous rocks, or nickel sulphide ores formed by replacement or magmatic injection (PIRSA Minerals, 2007). Sixty percent of Ni resources are of nickeliferous laterites and the rest of the sulphide. The nickel sulphide ores are associated with the platinum group metals (PGMs). There are approximately 30 minerals (Nickel Minerals, 2007) which contain Ni of which the most abundant is pentlandite. Its extraction and purification, and applications constitute a huge multi billion dollar industry. Its applications stretch into stainless steel, coinage, magnets, special alloys, painting and tinting, and laboratory uses (Nickel, 2007; Nickel Mineral Data, 2007).

An area in which Ni has found applications is the usage of thin films, which are essential in modern technologies and their optimization is the focus of continued growing research. Its application in thin films promotes the fabrication of nanostructured materials. By altering columnar inclination angle (β), and vapour incidence angle (α), different and varied properties of the films emerge which are characteristic for special applications. Other factors, which affect the film structure, include material and deposition conditions such as substrate temperature, angular distribution of the deposition flux, background gas pressure and composition, and flux energies. Deposition patterns of films could be normal, helical or zigzag. These patterns coupled with the listed factors herein influence degree of crystallization; and hence determine application of product.

In varying these factors, and controlling the column angle (β) and the flux angle (α) the density will vary in a predictable but not independently controllable or desirable manner. This direct dependence on flux angle (α) of both column angle (β) and film density restricts the range of thin film microstructure attainable. For example, a porous film with vertical microstructure would be forbidden. The limitation could be overcome by producing Ni films using glancing angle deposition (GLAD) (Robbie *et al*., 1997). This technique combines oblique deposition onto a flat substrate with computer control of substrate rotation, enabling the column angle and film porosity to be chosen independently by rotating the substrate in a controlled manner. With control of microstructure, material properties for optical, chemical, biological, mechanical, and electrical applications can be tailored to a much higher degree of accuracy.

Being a novel technique of thin film deposition, demonstrated applications are increasingly developed. Some already feasible usages include magnetic media high density information storage (Hsieh *et al.*, 1997), liquid crystal display technology (Robbie *et al*., 1999), photonic crystal (Kennedy et al., 2003), optical rotators, polarization beam splitters (Azzam, 1992), optical filters (Hodgkinson *et al*., 2000; Hodgkinson *et al*., 2000a; Kaminska *et al*., 2003). The technique is being explored for applications in the bioluminescence sensors, electroluminescent devices, optical transparent conducting films from pure metals, multistate electronic switches based on filamentary conduction, optical sensors that can detect and quantify various chemical and biological fluids, microsieves for entrapment of viruses, porous materials for growing biological tissues, chemical sensors, catalytic reaction surfaces, optical coatings, thermoelectric materials, quantum effect devices, field emitters, and solar cells.

Different analytical tools have been used in characterizing thin films (Murakami *et al*., 1996; Bueno-Core *et al*., 2004; Noh *et al*., 2000). One of such tool is the X-ray diffractometry. The XRD

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technique aids in finger printing phases present in an analyte by identifying and characterizing it. There is increasingly wide significance and promising applications of Ni thin films and the potential for those particularly produced from GLAD technique to gain strong industrial applications. This study thus employed X-ray diffractometry in understanding Ni thin films crystallization. It is anticipated that the generated knowledge will form basis for further applications of Ni thin films.

MATERIALS AND METHODS

Film preparation: Four different samples of Ni films (samples Ni0, Ni40, Ni80 and Ni70) were prepared by GLAD technique by evaporating Ni wire onto glass substrates in a vacuum chamber at pressures not greater than 10^{-7} mb at room temperature. Corning 1737 glass substrates were cleaned and attached to substrate holder in the chamber. The substrate was tilted and rotated as desired. The tilt gave the vapour incidence angle to the substrate normal. The rotation of the substrate was computer controlled to give the desired shape of the microstructure. Stepper motors controlled the deposition angle, (α tilt) and rotation of the substrate about an axis normal to its surface (ϕ rotation). A computer based feedback control system actuates the motors in accordance with parameterized programs of α and ϕ as functions of accumulated film thickness. By moving the substrate in a controlled way during deposition, the shape of the columns was accurately tailored on a nanometer scale. The columnar morphology was therefore directly related to the dynamically controlled angular coordinates (α, ϕ) of the vapour deposition source

Sample Ni0 was prepared with the glass substrate horizontal; with the vapour incident angle to the substrate normal being 0^o and the film thickness 300 nm. Sample Ni40 was prepared with the glass substrate tilted; with the vapour incident angle to the substrate normal being 40^o and the film thickness 200 nm from two turns of a 100 nm each. It was helical in microstructure with two rotations. Sample Ni80 was prepared with the glass substrate tilted; with the vapour incident angle to the substrate normal being 80^o and the film thickness 200 nm from two turns of a 100 nm each. It was also helical in microstructure with two rotations. Sample Ni70 was prepared with the glass substrate tilted; with the vapour incident angle to the substrate normal being 70^o and the film thickness 1000 nm from 10 zigzags. Its microstructure was zigzag.

X-Ray Diffraction Analysis: The X-ray diffractometer (Philips PW 3710 XRPD system) used for the analyses of the samples operated at 40 kV and 45 mA, with a Cu-Ka radiation and a graphite monochromator with PW 1877 Automated Powder Diffraction. A 2001 X'PERT Data Collector software package was used for capturing of phases present in the different samples. The X-rays were produced at the wavelengths $\lambda_1 = 1.54056$ Å and $\lambda_2 = 1.54439$ Å with λ_2 stripped off. Samples were scanned from 2° 2θ to 80° 2θ using a step size of 2θ = 0.02 and a time per step of 0.30 seconds and their diffractograms were recorded. A 2001 version of the Philips X'PERT graphics & Identify software package was used for qualitative identification of the minerals from both the data and patterns obtained by scanning. The interpreted results were compared with data and patterns available in the mineral Powder Diffraction File data book and search manual issued by the International Centre for Diffraction Data (2001) for confirmation.

RESULTS AND DISCUSSION

Figures 1 to 4 are the X-ray diffractograms of the samples, and Table 1 gives their peak properties according to peak angle, peak height and peak tip width. The ideal 100% RI peak for Ni is at 44.5 2θ, and other significant peaks for Ni identification are the 42% RI at 51.8 2θ, and 21% RI at 76.3 2θ. Sample Ni0 was crystallined with only the 100% RI peak very well defined. Sample Ni40 was crystallined with both the 100% and 42 % RI peaks very well defined. Whereas sample Ni70 was amorphous with not a single defined peak, sample Ni80 was poorly crystallined with its peaks not clearly defined.

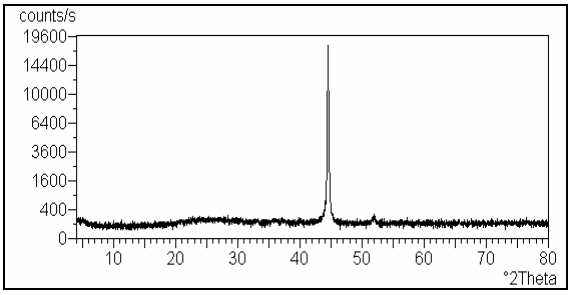

Fig 1: X-ray diffractogram of normal deposited Ni thin film (sample NiO)

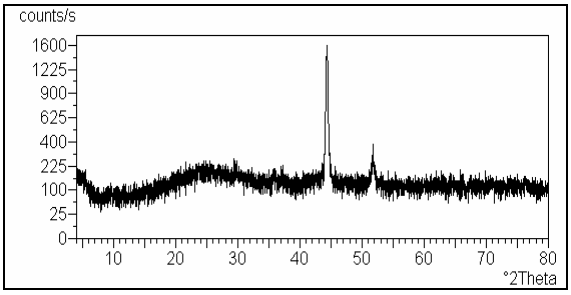

Fig 2: X-ray diffractogram of helical deposited Ni thin film (sample Ni40)

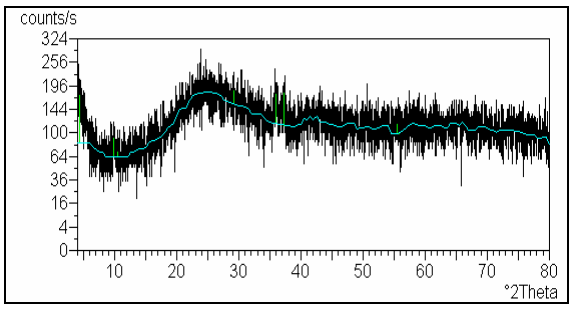

Fig 3: X-ray diffractogram of zigzag deposited Ni thin film (sample Ni70)

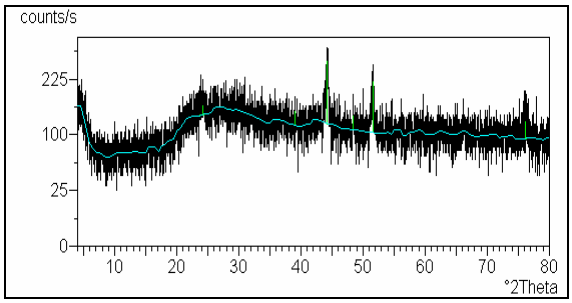

Fig 4: X-ray diffractogram of helical deposited Ni thin film (sample Ni80)

Table 1: Peak characteristics obtained from X-ray diffractometry of samples of Ni thin films

S/ No	Peak angle (2θ angle)	Relative intensity (%)	Miller indices (hk*l*)	Peak height (count s/s)	Peak tip width (2θ)
Ni0	44.5	100	111	1798.4	0.10
	51.9	42	200	161.9	0.16
Ni40	44.4	100	111	1440.9	0.18
	51.7	42	200	158.4	0.40
Ni70	-			-	-
Ni80	44.3	100	111	156.3	0.24
	51.8	42	200	113.9	0.20
	76.2	21	220	31.3	0.64

Using glancing angle deposition, porous chiral and zigzag Ni films with tailourable microstructure were fabricated. With this technique of growth during spinning of the substrate and growth while the substrate is stationary, column angle and azimuthal orientation can be controlled independent of film density. Film density is then specified by choosing the oblique angle of vapour flux incidence at the substrate. Helical and zigzag structures with modified geometry have been demonstrated.

In understanding the crystallinity of the Ni thin films, X-ray diffrctometry was employed. The X-ray diffractometry thus demonstrated that Ni thin film prepared by GLAD in which the glass substrate is tilted, and having a vapour incident angle to the substrate normal being 40^o and the film thickness 200 coupled with a helical microstructure; had higher crystallinity compared to that prepared normally. This GLAD technique therefore is suitable for the production of Ni thin films, and the characterization. It defines with the aid of X-ray diffractometry how thin films of high crystallinity could be produced with significant accuracy.

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REFERENCES

Azzam R. M. A. (1992) Chiral thin solid films: Method of deposition and applications. Applied Physics Letters 61 (26), 3118-3120

Bueno-Core G. E., Tejos M., Schrebler R., Klahn A. H. and Hill R. H. (2004) Nickel tropolonate complexes as precursors for the direct photodeposition of NiO thin films. Journal of Chilean Chemical Society 49 (3), 223-226.

Hsieh Y. C., Gadetsky S., Suzuki T., and Mansuripur M. (1997) Oblique sputtering of amorphous TbFeCo thin films on glass substrates and the effect of deposition angle on perpendicular magnetic anisotropy. Journal of Applied Physics 81 (8,) 3555-3560

Hodgkinson I. J., Lakhtakia A. and Wu Q. H (2000) Experimental realization of sculptured-thin-film polarization-discriminatory light-handedness inverters. Optical Engineering 39, 2831-2834.

Hodgkinson I. J., Wu Q. H., Thorn K. E., Lakhtakia A. and McCall M. W. (2000) Spacerless circular-polarization spectral-hole filters using chiral sculptured thin films: theory and experiment, Optics Communications, 184 (1-4), 57-66.

International Centre for Diffraction Data (2001) International Centre for Diffraction Data. Mineral Powder Diffraction File Databook. Pp 942.

Kaminska K., Brown T., Beydagghyan G. and Robbie K. (2003) Vacuum Evaporated Porous Silicon Photonic Interference Filters. Applied Optics 42, 4212-4219.

Kennedy S. R., Brett M. J., Miguez H., Toader O. and John S. (2003) Optical properties of a three-dimensional silicon square spiral photonic crystal. Photonics and Nanostructures 1, 37-42

Murakami Y., Kuratani N., Imai O. and Ogata K. (1996) Study on the crystallization of nickel films prepared by the ion beam and vapour deposited method. European Materials Research Society 1995 Spring Conference, Symposium E: Structures and properties of metalic thin films and multilayers, 275 (1-2) 61-63.

Nickel (2007) Nickel – Wikipedia, the free encyclopedia. http://en.wikipedia.org/wiki/Nickel. Accessed 27 April 2007.

Nickel Minerals (2007) Category: Nickel minerals. Wikipedia, the free encyclopedia. http:/en.wikipedia.org/wiki/Category:Nickel_minerals. Accessed 28 April 2007.

Nickel Mineral Data (2007) Nickel mineral data. http://webmineral.com/data/Nickel.shtml. Accessed 28 April 2007.

Noh H. Y., Lee K. H., Cul X. X. and Choi C-S. (2000) The composition and structure of TiNi thin film formed by electron beam evaporation. Scripta Materialia 43(9) 847-852.

PIRSA Minerals (2007) Nickel. Mineral Resource Potential – Nickel. http://www.pir.sa.gov.au/pages/minerals/commodity/nickel.htm. Accessed 27 April 2007.

Robbie K., Hnatiw A. J. P., Brett M. J., MacDonald R. I. and McMullin N. J. (1997) Inhomogeneous Thin Film Optical Filters Fabricated Using Glancing Angle Deposition. Electronics Letters 33(14) 1213-1214.

Robbie K. Broer D.J. Brett M.J (1999) Chiral nematic order in liquid crystals imposed by an engineered inorganic nanostructure. Nature 399, 764-766.