ELSEVIER

Available online at www.sciencedirect.com

SciVerse ScienceDirect

Physics Procedia 23 (2012) 61 – 64

Physics Procedia

Asian School-Conference on Physics and Technology of Nanostructured Materials (ASCO-NANOMAT 2011)

# Micromorphology and spectroscopic ellipsometry of Ni(100) crystal surface

T.A. Gavrilova[a,*], V.V. Atuchin[b], V.N. Kruchinin[c], D.V. Lychagin[d]

[a]*Laboratory of Nanodiagnostics and Nanolithography, A.V. Rzhanov Institute of Semiconductor Physics, SB RAS, 13 Lavrentieva ave., Novosibirsk 630090, Russia*

[b]*Laboratory of Optical Materials and Structures, A.V. Rzhanov Institute of Semiconductor Physics, SB RAS, 13 Lavrentieva ave., Novosibirsk 630090, Russia*

[c]*Laboratory of Ellipsometry of Semiconductor Materials and Structures, A.V. Rzhanov Institute of Semiconductor Physics, SB RAS, 13 Lavrentieva ave., Novosibirsk 630090, Russia*

[d]*Department of Materials Technology, Tomsk State University of Architecture and Building, 2, Solyanaya Square, Tomsk 634003, Russia*

**Abstract**

Dispersion of refractive index and extinction coefficient of nickel crystal surface (100) has been determined with spectroscopic ellipsometry over spectral range λ ~ 250–1030 nm. The crystal surface (100) at a first stage was mechanically polished and then treated by electro-polishing to remove the damage layer.



*Keywords*: nickel; ellipsometry; optical constant

## 1. Introduction

Nickel metal thin films are widely used in electrochemistry, microelectronic devices, solar energetics and nanotechnology [1-3]. Electrical conductivity, structural and optical parameters of nanometric nickel film are strongly dependent on film thickness [4-6]. In integrated optics the nanometric nickel films are

* Corresponding author. Tel.: +7-383-330-9082; fax: +7-383-333-1080.
*E-mail address*: gavr@thermo.isp.nsc.ru.


doi:10.1016/j.phpro.2012.01.016

used as a source for doping $LiNbO_3$ substrate by thermal diffusion [7,8]. In comparison with Ti- or H-doped and ion-implanted waveguides, the Ni:$LiNbO_3$ layers possess lower anisotropy of refractive index increases with evident benefits for creation of integrated devices insensitive to lightwave polarization [9-14]. Because effective refractive indices of the waveguide modes are strongly dependent on the nickel profile over doped layer, precise control of Ni film thickness $h$ is needed in the thickness range $h \sim 10\text{-}50$ nm. All these applications request for precise control of nickel film parameters, thickness in particular.

Ellipsometry can be successfully applied for noncontact and nondestructive determination of the thickness of dielectric and semi-transparent metal film when optical constants of the material are well defined [4,15,16]. Regrettably, noticeable scattering was found for optical constants reported earlier in literature for nickel films and crystals. Present study is aimed to develop the technology of nickel crystal grow. Optical parameters of the grown nickel single crystal will be determined with spectroscopic ellipsometry (SE) to have a basis for comparison with those of thin films.

## 2. Experimental

Nickel single crystal was grown from machined polycrystalline blanks (99,995%) by horizontal Bridgman method in graphite mold. Laue X-ray back reflection method was used for crystal orientation. Specimens were sliced from the single crystal by means of an acid saw. The specimens were electro polished in chromic anhydride and orthophosphoric acid saturated solution at 20 V and room temperature for 2-3 min to remove the surface roughness created by the acid saw operation. Quality and crystallographic orientation of surface polish was checked by X-ray, optical and scanning electron microscopy. Detailed description of the single crystal metal sample preparation can be found elsewhere [17,18]. The specimen with the smoothest polished surface was selected for ellipsometry measurements.

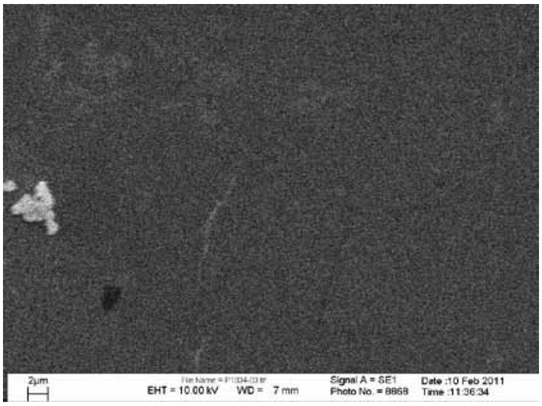
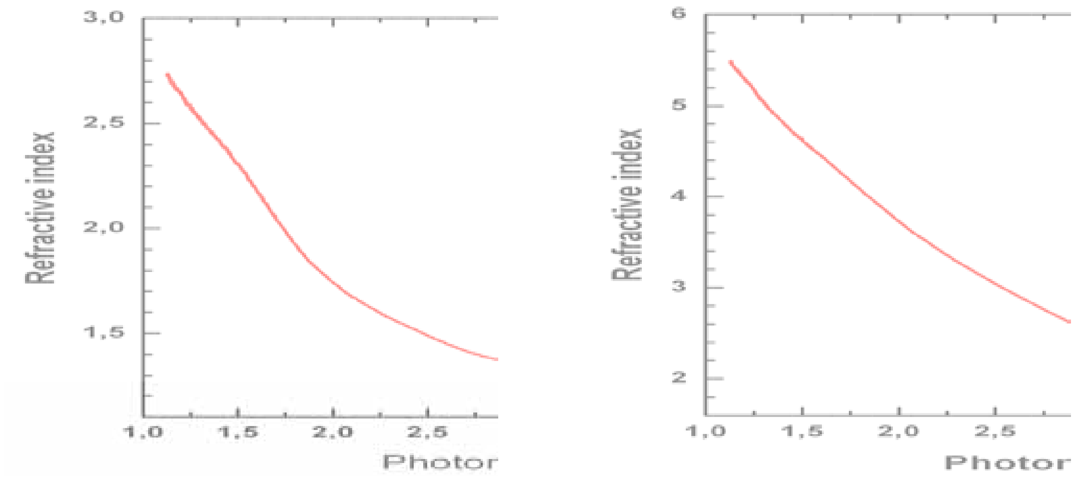



Fig.1. SEM image of electro-polished Ni(100) surface. A dust particle is visible in the left side of the image

Micromorphology of Ni(100) polished surface was evaluated with scanning electron microscopy (SEM). Spectral dependencies of refractive index $n(\lambda)$ and extinction coefficient $k(\lambda)$ were determined with the help of spectroscopic ellipsometry (SE). Ellipsometric angles $\Psi$ and $\Delta$ were measured as a function of $\lambda$ using a EFFIPS-1771 SA ellipsometer [19]. The instrumental spectral resolution was 2 nm, exceed 20 s and the angle of incidence of light beam on the sample was 70°. The ellipsometry parameters $\Psi$ and $\Delta$ are related to the complex Fresnel reflection coefficients by well-known equation [20,21]. To calculate the dependencies of refractive index $n(\lambda)$ and extinction coefficient $k(\lambda)$, the experimental data were processed using the model of air-homogeneous isotropic substrate [21-25].




a b

Fig.2. Dispersion of (a) *n* and (b) *k* of Ni(100) crystal surface

## 3. Results and Discussion

In Fig. 1 the SEM pattern is shown for prepared nickel surface. High quality of the surface is evident. There is no developed system of etching defects that is valuable for robust optical measurements. Dispersion of optical parameters is shown in Fig. 2. The *n* and *k* decrease with a photon energy increase. Local maximum is found, however, for *n* at photon energy of 4.0 – 4.2 eV. An increase of *k* is observed for photon energy above 4.0 eV. These curves obtained for nickel single crystal can be used as a basis for comparison with optical parameters of nickel thin films fabricated with different techniques.

## 4. Conclusions

A technology of nickel crystal growth and optical surface preparation is developed to have highly reproducible metal surface characteristics. Now optical parameters of nickel are accurately measured over wide spectral range and can be used as a basis for precise determination of thickness of semi-transparent nickel films.

## References

[1] Khyzhun O, Sygellou L, Ladas S. Interfacial oxidation of ultrathin nickel and chromium films on yttria-stabilized zirconia. *J Phys Chem B* vol. 2005;**109**:2302-6.

[2] Adsten M, Joerger R, Järrendahl K, Wäckelgård E. Optical characterization of industrially sputtered nickel-nickel oxide solar selective surface. *Solar Energy* 2000;**68**:325-8.

[3] Yin Y, Pan Y, Rubanov S, Bilek MMM, McKenzie DR. Sputtered nanocrystalline nickel thin films for solar thermal energy applications. *Nanosci Nanotechnol Lett* 2009;**1**:1-5.

[4] Kamineni VK, Raymond M, Bersch EJ, Doris BB, Diebold AC. Optical metrology of Ni and NiSi thin films used in the self-aligned silicidation process. *J Appl Phys* 2010;**107**:093525.

[5] Lee Han-Bo-Ram, Bang Sung-Hwan, Kim Woo-Hee, Gu Gill Ho, Lee Young Kuk, Chung Taek-Mo, Kim Chang Gyoun, Park Chan Gyung, Kim Hyungjun. Plasma-enchanced atomic layer deposition of Ni. *Jpn J Appl Phys* 2010;**49**:05FA11.

[6] Xu J, Shao T, Guo J. Effect of processing conditions on microstructure and electrical characteristics of Ni thin films. *Vacuum* 2010;**84**:478-82.

[7] Wei Pei-Kuen, Wang Way-Seen. A TE-TM mode splitter on lithium niobate using Ti, Ni, and MgO diffusions. *IEEE Photon Technol Lett* 1994;**6**:245-248.

[8] Cheng Rei-Chin, Chen Wei-Lin, Wang Way-Seen. Mach-Zehnder modulators with lithium niobate ridge waveguides fabricated by proton-exchange wet etch and nickel indiffusion. *IEEE Photon Technol Lett* 1995;**7**:1282-4.

[9] Atuchin VV, Ziling CC, Shipilova DP, Beizel NF. Crystallographic, ferroelectric and optical properties of $TiO_2$-doped $LiNbO_3$ crystals. *Ferroelectrics* 1989;**100**:261-9.

[10] Atuchin VV, Ziling CC, Savatinova I, Armenise MN, Passaro VMN. Waveguide formation mechanism generated by double doping in ferroelectric crystals. *J Appl Phys* 1995;**78**:6936-9.

[11] Savatinova I, Ziling CC, Atuchin VV. Metastable states in proton exchanged layers H:$LiMO_3$ (M = Nb, Ta). *Opt Mater* 1999;**12**:157-162.

[12] Atuchin VV. Causes of refractive indices changes in He-implanted $LiNbO_3$ and $LiTaO_3$ waveguides. *Nucl Instr Meth Phys Res B* 2000;**168**:498-502.

[13] Kalabin IE, Grigorieva TI, Atuchin VV. Formation and decay of high temperature phase in $H_xLi_{1-x}NbO_3$ layers. *Opt Mater* 2003;**23**:281-4.

[14] Kalabin IE, Shevtsov DI, Azanova IS, Taysin IF, Atuchin VV, Volyntsev AB, Shilov AN. Quenching effects on crystallographic and optical properties of H:$LiNbO_3$ layers. *J Phys D: Appl Phys* 2004;**37**:1829-33.

[15] Jansen GJ, Sorensen JM, Kearney RJ. Ellipsometric determination of optical constants of metals; a contribution to reflectivity standards for ore microscopy. *Econom Geol* 1969;**64**:325-8.

[16] Kirilova MM, Knyazev YuV, Kuzmin YuI. Spectroellipsometric study of phase transitions in d and f metals in ultrahigh vacuum. *Thin Solid Films* 1993;**234**:527-30.

[17] Koneva NA, Starenchenko VA, Lychagin DV, Trishkina LI, Popova NA, Kozlov EV. Formation of dislocation cell substructure in face-centered cubic metallic solid solutions. *Mater Sci Eng A* 2008;**483**: Sp. Iss. SI 179-83.

[18] Teplyakova LA, Bespalova IV, Lychagin DV. Spatial organization of deformation in aluminum [1-12] single crystals in compression. *Phys Mesomechanics* 2009;**12**:166-74.

[19] Rykhlitski SV, Spesivtsev EV, Shvets VA, Prokopiev VYu. Spectroscopic ellipsometry complex "Ellipse-1771 SA". *Instrum Experim Techniques* 2007;(2):160-1 (in Russian).

[20] Shvets VA, Aliev VSh, Gritsenko DV, Shaimeev SS, Fedosenko EV, Rykhlitski SV, Atuchin VV, Gritsenko VA, Tapilin VM, Wong H. *J Non-Cryst Solids* 2008;**354**:3025-33.

[21] Atuchin VV, Ayupov BM, Kochubey VA, Pokrovsky LD, Ramana CV, Rumiantsev YuM. Optical properties of textured $V_2O_5$/Si thin films deposited by reactive magnetron sputtering. Opt Mater 2008;**30**:1145-8.

[22] Ramana CV, Utsunomiya S, Ewing RC, Becker U, Atuchin VV, Aliev VSh, Kruchinin VN. Spectroscopic ellipsometry characterization of the optical properties and thermal stability of $ZrO_2$ films made by ion-beam assisted deposition. *Appl Phys Lett* 2008;**92**:011917.

[23] Atuchin VV, Kruchinin VN, Kalinkin AV, Aliev VSh, Rykhlitskii SV, Shvets VA, Spesivtsev EV. Optical properties of the $HfO_{2-x}N_x$ and $TiO_{2-x}N_x$ films prepared by ion beam sputtering. *Opt Spectr* 2009;**106**:72-77.

[24] Atuchin VV, Kalinkin AV, Kochubey VA, Kruchinin VN, Vemuri RS, Ramana CV. Spectroscopic ellipsometry and x-ray photoelectron spectroscopy of $La_2O_3$ thin films deposited by reactive magnetron sputtering. *J Vac Sci Technol A* 2011;**29**:021004.

[25] Ramana CV, Mudavakkat VH, Bharathi KK, Atuchin VV, Pokrovsky LD, Kruchinin VN. Enhanced optical constants of nanocrystalline yttrium oxide thin films. *Appl Phys Lett* 2011;**98**:031905.