A STRUCTURE AND PHYSICAL PROPERTIES OF Ni FILMS IN METASTABILE STATES

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ABSTRACT

There are presented the results of investigations of the influence of deposition rate and pressure of orifice gas on the structure, physical properties and thermal stability of nickel films obtained by the modified method of the three-electrode ion-plasmous sputtering (technique IPS).

X-ray analysis and estimation of the size (L) of coherent-scattering regions (CSR) showed that in the as-deposited films which obtained with low energy of sputtered atoms and low deposition rate (~ 85 pm/s) there is formed a mixture of FCC Ni (L = 5 nm), traces of HCP nickel (L = 9 nm) and oxide of nickel NiO. Increase of energy of sputtered atoms by a decrease of pressure of orifice gas from 120 to 53 or 16 mPa leads to the formation at as-deposited films a mixture of phases FCC nickel and traces of oxides. Moreover, almost double increase of deposition rate leads to an increase the size of the CSR on ~20%. The heating of films of Ni with a mixture of FCC and HCP phases to 300-320 °C results in transformation of HCP in FCC. Freshly deposited films with the structure of FCC Ni are undergoing structural changes at heating above 350-450 °C. These changes are linked obviously with the recrystallization. The size of the CSR for FCC Ni is increased almost in 2 times after heating.

It is shown that energy of activating (E_A) calculated by the method of Kissinger in the case of disintegration of HCP Ni exceeded from E_A of beginning of recrystallization almost in three times and attains ~21000 K. In addition, it's shown that increase of deposition rate brings to rise of activation energy of beginning recrystallization and indicates the formation of more stable structure in the films. Analysis of the demagnetization curves of the films of nickel showed anisotropy of magnetic properties. Hysteresis only is detected in a parallel field to the film of Ni. The coercive force does not exceed 200 A/m.

Key words: nickel films, sputtering, coherent-scattering region, FCC and HCP nickel

INTRODUCTION

The extreme sensitivity of the technological conditions of sputter deposited films allows to vary the properties of these films and to observe phenomena which are not recorded in the bulk samples.

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It is known that high kinetic energy of the condensed atoms allows at forming of films to get them in the nonequilibrium state. For the increase of energy of the condensed atoms the electrostatic accelerating of Ar ions can be used [1]. It was before shown that kinetic energy of the condensed atoms at the use of method of modernized ion-plasmous sputtering it is possible to change from 100 to 200 eV changing pressure from 53 to 13 mPa [2, 3]. In this case the effective cooling rate of films is connected with the relaxation time of individual atom on substrate and in theory estimated as 10^{12}- 10^{14} °C/s [4]. That, in this case there is a "quenching from the vaporous state" (QVS).

Therefore interestingly it was to find out how energy of the deposited atoms and rate of deposition influences on structure and physical properties of films. As an object films of nickel were chosen.

METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

There was used the modified method of the three-electrode ion-plasmous sputtering [1]. The vapor deposition of the films was carried out under the conditions, resulted in a *table 1.*

Table 1 - Conditions of sputter deposited Ni films

	Ni1	Ni2	Ni3	Ni4	Ni5	Ni6	Ni7
U, (kV)	-2	-2	-2	-2	-2	-2	-2
I_{an}, (A)	2	0.8	0.8	1	1	2	0.8
P_w (mPa)	53	16	16	120	53	53	16
φ, (eV)	100	200	200	20	100	100	200
t_{dep}, (min)	24	24	28	13	24	18	30
d, (nm)	200	115	140	79	103	158	139
η, (pm/s)	140	80	80	101	72	147	77

Where U – is a target voltage; I_{an} – is anode current; P_{Ar} – is a pressure of orifice gas (Ar); t_{dep} – is a time of sputter deposition; d - is a thickness of films; η - is a rate of deposition; φ - is estimated kinetic energy of the condensed atoms [2, 3].

The deposition was performed under equal conditions simultaneously onto the Sitall substrates and the freshly cleaved single crystals of sodium chloride. The films deposited onto NaCl were separated by dissolving the salt in distilled water and used for studying the structure and phase composition by the X-ray diffraction (XRD) and transmission electron microscopy technique (TEM). The physical properties and thermal stability were studied using the films deposited onto Sitall. The sheet resistance was measured by the four point technique upon continuously heating the film in vacuum 10 mPa. The structure and compositions of the initial and annealed films were studied by the X-ray diffraction patterns obtained with RKD cameras using the CoK_α radiation. The coercive force H_c was measured on a vibration magnetometer in the maximal magnetizing field 1 T, attached parallel and athwart surfaces of film.

RESULTS AND DISCUSSION

Figure 1 presents an XRD patterns from Ni4 films in as deposited state and after heat treatment.

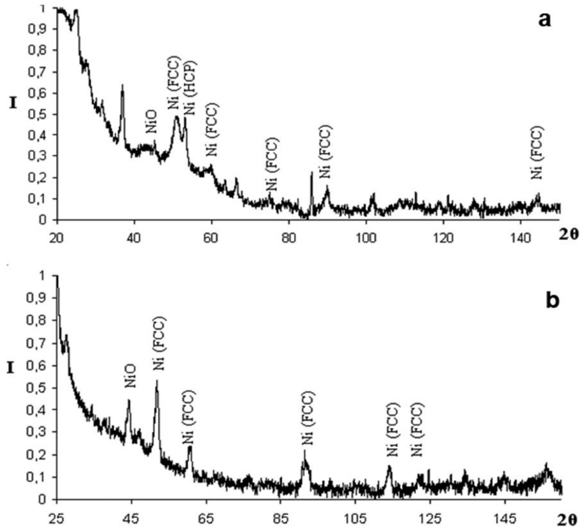

Fig. 1 – X-ray diffraction patterns from Ni4. Curve (a) refer to the as deposited state and curve (b) to the film annealed at 873 K for 5 min.

Analysing these XRD patterns and XRD patterns of other samples one can see that in the fresh deposited films which obtained with low energy of sputtered atoms (~ 20 eV) and low deposition rate (~ 100 pm/s) there is formed a mixture of FCC Ni (L = 5 nm), traces of HCP nickel (L = 9 nm) and oxide of nickel NiO. The size (L) of coherent scattering regions (CSR) was appraised on the formula of Selyakov-Sherer formula. Increase of energy of sputtered atoms by a decrease of pressure of orifice gas from 120 to 16-53 mPa leads to the formation at freshly deposited films a mixture of phases FCC nickel and traces of oxides. Moreover, almost double increase of deposition rate leads to an increase the size of the CSR on ~20%. The heating of films of Ni with a mixture of FCC and HCP phases to 300-320 °C results in transformation of HCP in FCC (*Fig. 1b*).

Figure 2 exhibits temperature variation of resistivity of Ni films and temperature intervals of structural transformations. In figure 1-2 one can see that the heating of films of Ni with a mixture of FCC and HCP phases to 300-320 °C results in transformation of HSP in FCC. Freshly deposited films with the structure of FCC Ni are undergoing structural changes at heating above 420-450 °C. These changes are linked obviously with the recrystallization. The size of the CSR for FCC Ni is increased almost in 2 times after heating.

It is shown that energy of activating (E_A) calculated by the method of Kissinger in the case of disintegration of HSP Ni exceeded from E_A of beginning of recrystallization almost in three times and attains ~21000 K.

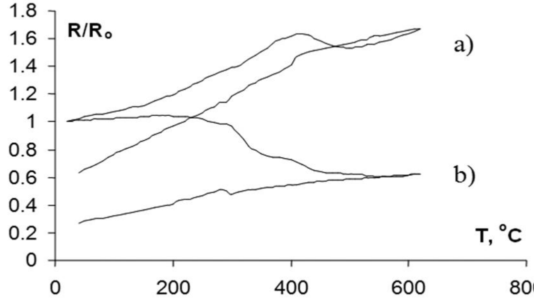

Fig. 2 – Temperature variation of resistivity of films. Curve (a) refer to the Ni4 with a mixture of FCC and HCP phases and curve (b) – Ni5 with a FCC phase

In addition, it's shown that increase of deposition rate brings to rise of activation energy of beginning recrystallization and indicates the formation of more stable structure in the films.

Figure 3 presents an demagnetization curves of the Ni6 films in as deposited state.

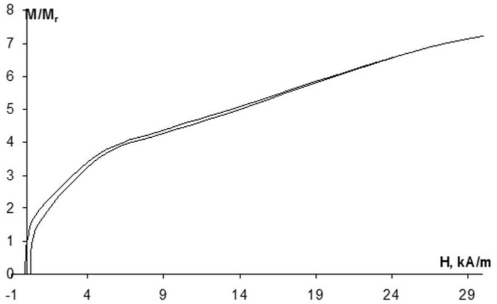

Fig. 3 – Demagnetization curves of the Ni6 films in as deposited state.

Analysis of the demagnetization curves of the films of nickel showed anisotropy of magnetic properties. Hysteresis only is detected in a parallel field to the film of Ni with a FCC phase. The coercive force does not exceed ~200 A/m. Hysteresis don't detected in Ni4 with a mixture of FCC and HCP phases in as deposited state.

CONCLUSIONS

Studies have shown that the choice of conditions of films production is given by possibility to manage size of coherent scattering regions (CSR) and activation energy of phase transitions depending on the assigned task.

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