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Structure, surface morphology and electrical properties of evaporated Ni thin films: Effect of substrates, thickness and Cu underlayer

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ABSTRACT

Series of Ni thin films have been deposited by thermal evaporation onto glass, Si(111), Cu, mica and Al₂O₃ substrates with and without a Cu underlayer. The Ni thicknesses, t , are in the 4 to 163 nm range. The Cu underlayer has also been evaporated with a Cu thickness equal to 27, 52 and 90 nm. The effects of substrate, the Ni thickness and the Cu underlayer on the structural and electrical properties of Ni are investigated. Rutherford Backscattering Spectroscopy was used to probe the Ni/Substrate and Ni–Cu underlayer interfaces and to measure both Ni and Cu thicknesses. The texture, the strain and the grain size values were derived from X-ray diffraction experiments. The surface morphology is studied by means of a Scanning Electron Microscope. The electrical resistivity is measured by the four point probe. The Ni films grow with the <111> texture on all substrates. The Ni grain sizes D increase with increasing thickness for the glass, Si and mica substrates and decrease for the Cu one. The strain ϵ is positive for low thickness, decreases in magnitude and becomes negative as t increases. With the Cu underlayer, the growth mode goes through two phases: first, the stress (grain size) increases (decreases) up to a critical thickness t_c , then stress is relieved and grain size increases. All these results will be discussed and correlated.

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1. Introduction

The Ni thin films have been the subject of a lot of studies. Lately the interest is focused for films with thickness in the nanometer range. It is found that the physical properties of Ni strongly depend on the method and conditions of deposition, on the thickness, the substrate and the underlayer. Among recent work on Ni, one may cite the paper by Zou et al. [1] where the authors did a comparative study of the physical properties of Ni on SiO₂/Si(100) and Ni on anodic aluminum oxide; the former being a continuous film while the latter has a network structure. Yu et al. [2] made a comparison between the Ni characteristics deposited on SiO₂/Si(100) by oblique angle sputtering and conventional sputtering. The structure and magnetic properties were also investigated for DC magnetron sputtered Ni films onto glass substrate [3], the authors showed that the 15 nm thick film presents a large amount of amorphous structure which they believe is responsible for the decrease of the magnetoresistance in this thin sample. Some interesting phenomena have been reported in Ni thin films. The Anomalous Hall Effect (AHE) has attracted a lot of attention lately. The AHE phenomenon has been studied in magnetron sputtered polycrystalline Ni thin film on oxidized Si substrate as a function of Ni thickness (4 to 200 nm) [4]

and also in a 10 nm thick RF sputtered Ni film [5] where the authors correlate the behavior of the Hall resistance and the extraordinary Hall coefficient with the film thickness and morphology. The other interesting feature observed in Ni films is the transition of the magnetization easy axis from in-plane to out-of-plane as a function Ni thickness [6]; this was observed in Ni/Cu and was attributed to a stress induced anisotropy. Ni films were studied not only as a single film but also as part of a structure. For instance the physical properties of Ni/Cu/p-Si Schottky diodes prepared by liquid phase epitaxy have been investigated [7]. A lot of work on Ni based multilayers have been reported, e.g. the stress in electrodeposited Ni/Cu multilayer film on stainless steel substrate has been investigated, it was found that Ni/Cu multilayers are characterized by high hardness and strength and could be used in many applications such as in magnetic devices and micro-actuators [8].

In the present work, we have studied the structural and electrical properties of two Ni based systems: the evaporated Ni thin films deposited on four different substrates (glass, Si, mica, Cu and Al₂O₃) and with thickness in a nanometer range (9 to 163 nm) and the Ni/Cu/Substrates where the Cu thicknesses are set to 27, 52 and 90 nm and the Ni thickness ranges from 4 to 67 nm. The objectives of the present study are: (i) to investigate the effect of different substrates on the physical properties of the evaporated Ni thin films in this thickness range (9 to 163 nm) and (ii) to find out how these properties are modified when

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Ni is grown with Cu underlayer. We believe also that the present study might be useful to understand in part the behavior of Ni in a multilayer system. Note also that the structural properties can affect the magnetic properties of these Ni films; the stress may, for instance, induce a magnetic anisotropy through the inverse magnetostriction effect. The magnetic properties of these systems will be published later on. The experimental methods and the deposition conditions are shown in Section 2. In Section 3, we show and discuss the results pertaining to the study of the state of the interfaces, the structure, the surface morphology and the electrical resistivity. A conclusion is given in Section 4.

2. Experimental methods

The Ni thin films have been deposited by thermal evaporation onto five substrates: glass (soda-lime-glass), Si(111), Cu, mica and Al_2O_3 (Corundum) from a 99.99% purified Ni powder. The Ni thickness ranges from 9 to 163 nm. The five substrates have been chosen because of the different physical properties they present. From the structural point of view, these are amorphous (glass), polycrystalline (mica, Cu and Al_2O_3) and single crystal (Si) materials. For the electrical properties, we do have insulator, conductor and semiconductor. We believe that the different properties of these substrates may induce different behaviors of the deposited Ni thin films. The aim of this work is indeed to find out and ultimately to explain the induced properties of these Ni thin films in this nanometric thickness range. The deposition rate for Ni for this first system is between 0.2 and 0.9 Å/s. For the second system, using the same substrates as above (glass, Si(111), Cu and mica), we first evaporated a Cu underlayer and then without breaking the vacuum, we deposited the Ni thin film with different thicknesses. Pressure was about 3.8×10^{-7} mbar before evaporation; during deposition, it rises to 10^{-6} mbar. The substrates were not heated during the evaporation. In order to avoid oxidation or any other contaminations, the samples were kept under vacuum until all the characterization experiments were done. Rutherford Backscattering Spectroscopy (RBS) was used to probe the Ni/substrate and Ni–Cu underlayer interfaces and to measure both Ni and Cu thicknesses. The RBS experiments were done using 2 MeV He^+ ions delivered by a 3.75 MV Van de Graaf accelerator, the backscattering angle is equal to 20° . The structural properties (texture, lattice constant and grain sizes) were inferred from the X-ray diffraction (XRD) method working in the θ – 2θ mode with a wavelength $\lambda = 1.54$ Å. The surface morphology is investigated by means of a Scanning Electron Microscope (SEM). The SEM operating voltage was fixed to 20 kV for four samples (Ni/glass with $t = 50$ and $t = 163$ nm and the 14 nm thick Ni/Cu/glass and Ni/Cu/Si) and to 10 kV for the other ones. The electrical resistivity is derived from the sheet resistance which was measured by the four point probe.

3. Results and discussions

3.1. The Ni/Substrate system

RBS experiment was carried out to measure the film thickness and to probe the substrate–film interface. Examples of RBS spectra are shown in Fig. 1a for Ni (50 nm)/Si. The simulated spectra (solid lines) are obtained using the well-known RUMP code. The contributions of different elements of the film and the substrates are indicated in the figure. We can see that the different peaks are well separated, indicating that there is no interdiffusion at the interface between Ni and the substrates. From the fit of the experimental spectra and the simulated curves, we have inferred the film thicknesses. For this first system, i.e., Ni on different substrates, the Ni thicknesses, t , are equal to 9 ± 1 nm, 42 ± 3 nm, 50 ± 3 nm and 163 ± 5 nm.

X-ray diffraction experiments were performed on all samples. Examples of X-ray diffraction spectrum are shown in Fig. 2, for a 163 nm thick Ni/glass (Fig. 2a), Ni/Si (Fig. 2b) samples. For all samples, we observed a peak at $2\theta = 44.61^\circ$, which has been identified as the (111)

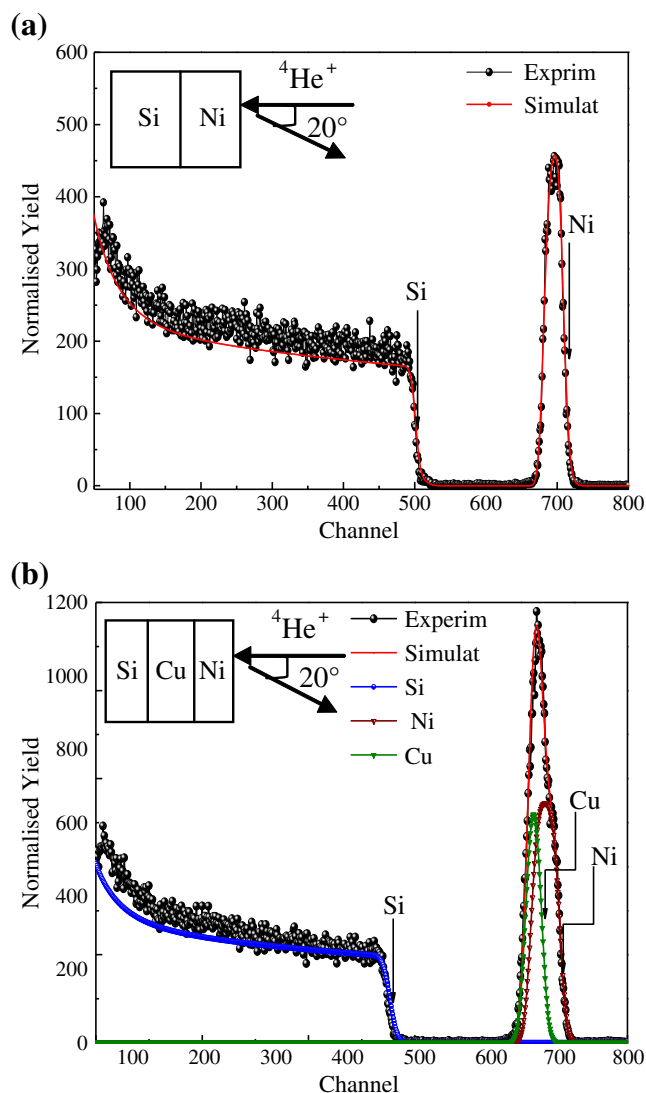


Fig. 1. RBS spectra for (a) Ni (50 nm)/Si and (b) Ni (67 nm)/Cu (52 nm)/Si. The points are experimental data; the solid line is the simulated spectra.

peak of the face centered cubic (fcc) Ni. As the thickness of the Ni film increases, the intensity of the (111) peak increases and other peaks of lower intensities are also seen, such as the (200) at $2\theta = 51.81^\circ$, the (220) at $2\theta = 76.53^\circ$, the (311) at $2\theta = 93.45^\circ$ and the (222) at $2\theta = 98.35^\circ$. The peak corresponding to the substrate, i.e. Si(311) peak, is also indicated in the figure. We have computed the intensity ratios of the peaks and compared these ratios to those of Ni powder. We infer from these spectra that the Ni thin films grow with the $\langle 111 \rangle$ texture for all substrates. Note however that for the Ni/Si, the intensity ratios are somewhat close to the Ni powder ones indicating a very weak $\langle 111 \rangle$ texture or no texture at all. While for the other substrates, there is a clear $\langle 111 \rangle$ texture. The $\langle 111 \rangle$ texture seems to be the most encountered one for Ni as reported in the literature. Indeed, it is found in DC magnetron sputtered Ni on $\text{SiO}_2/\text{Si}(100)$ and Al_2O_3 substrates with t in the 80 to 260 nm range [1] and in Ni grown on Al_2O_3 by chemical vapor deposition [9,10]. The $\langle 111 \rangle$ texture was also observed in 5 nm thick DC magnetron sputtered Ni/Si films [11], in RF magnetron sputtered Ni films [5]; in evaporated Ni/Si, Ni/Cu and Ni/glass [12,13]. In fact, in most cases the $\langle 111 \rangle$ is observed in Ni elaborated by different methods and on several substrates [14–21]. However, other textures were also reported for Ni, such as the $\langle 100 \rangle$ texture in electron beam evaporated Ni/ SiO_2/Si films [5]. The $\langle 220 \rangle$ and the $\langle 200 \rangle$ textures were observed in a 60 nm thick Ni [22,23].

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