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CHARACTERIZATION OF ANNEALED NI/CU MULTILAYERS ON SI(100)

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Abstract: In this work results of magnetic and structural measurements of annealed Cu/Ni multilayers, with different thicknesses of Ni and Cu prepared on (100) polycrystalline Si substrates, have been presented. Samples were obtained by the RF (DC) magnetron sputtering method and after deposition they were heated from 150°C to 350°C. The nanostructure of annealed Cu-Ni multilayers were analyzed using X-ray diffraction (XRD) and high-temperature XRD (HT-XRD). The annealing times of the multilayers varied from 0 to 180 minutes in vacuum chamber. The existence of satellite peaks in the XRD patterns showed that the multilayer films have superlattice structures.

Keywords: electrodeposition, Ni/Cu multilayer, ultra thin film, annealing

1. Introduction

Systems of thin metallic multilayers have been often used in magnetic recording and reading. In order to achieve a higher density of information packing, what is followed by smaller size of indicators, multilayers' borders contribute in the whole volume of the multilayer system. Therefore, the physical state of the interface has a significant effect on electric, magnetic and structural properties of the whole system. Recently, nanostructured materials such as, compositionally modulated alloys, multilayers have become widely studied. The first interest was to research magnetic and electrical properties (GMR effect) with the secondary goal of improving the tribological of these surfaces [1,2]. The mechanical properties of these surfaces have been also studied [3-6]. In spite of some limitations, like conducting substrates, the electrochemical methods has been used for preparation of such multilayer as Ni/Cu [7], NiCo/Cu [8], Co/Cu [9], NiFe/Cu[10], Co/Pt [11], in past decades.

In this paper, we report on the growth of Ni/Cu multilayers onto Si monocrystalline substrates by sputtering method. We show some polarization investigations of Si(100) substrates, paying attention to the establishing of proper deposition potentials for the Ni and Cu sublayers. We present the nanostructural characterization of the multilayers by high angle X-ray diffraction (XRD) and high temperature X-ray diffraction (HT-XRD). We also show how the structural properties depend on the total thickness of the deposited films and different annealing temperatures. Annealed Ni/Cu multilayers on Si(100) substrate are investigated. The main purpose of present work is to study of Ni/Cu multilayer when it was annealed. Finally we present the influence of bilayer and sublayer (Ni and Cu) thicknesses on the magnetic properties of multilayers.

2. Experimental details

Alternate layers of copper and nickel of varying thicknesses were deposited on silicon substrates at different substrate temperatures using an RF(DC) magnetron sputtering system. Si monocrystalline wafers with (100) orientation were used as substrates for deposition. Before loading the samples into evaporation chamber, their surfaces were refreshed in diluted HF and cleaned by the standard procedures used in microelectronics technology. The Cu and Ni targets were found to have a purity of 99.94% or greater and were deposited onto the substrate with a mass flow rate of 20.4 cm^3 min^{-1}.

Typically, Ni/Cu multilayers were deposited under a base pressure of ~10^{-6} torr and Ar gas pressure of 60 mbar. The substrate to target distance was 0.054 m. Equal number of Cu and Ni layers were deposited in all multilayer samples. In general, deposition rates of 1.0 and 1.6 nm^{-1} for Cu and Ni targets, respectively, were used in the experiments. The film thickness was determined by a quartz crystal monitor. We employed a Philips-X, Pert diffractometer with Cu-Kα radiation to carry out the X-ray diffraction experiments. X-ray diffraction (Cu-Kα radiation, λ = 0.15405 nm, Bragg-Brentano geometry) method was used for investigation of structure and phase composition transitions. The surface morphology was investigated by AFM. Magnetic properties of samples were also measured with MFM.

3. Results and discussion

The MFM and AFM pictures show distributed magnetic domains and topography of the sample surfaces, as depicted in Fig.1a and 1b for Ni/Cu multilayer on Si (100) substrate, respectively.

MFM was operated in a non-contact, dual-pass lift-mode technique with commercially available, CoCr-coated Si MFM tips. The tips were perpendicularly magnetized. The scan height was 5 μm. We show the result in Fig.1a. In this image, bright and dark areas can be regarded as corresponding to the position of inner (up) down domains. The periodicity or the width of the domains depends on the irradiation dose, ranging about 2–4 μm. We note that measurements on different magnetic samples indicate that the distance between the tip apex and the effective position of the magnetic moment in the tip depends on the magnetic properties of the measured surface and therefore the calculated magnetic moment from MFM data should be taken as a rough estimate only.

Surface topographical characterization gives significant additional information about the surface of samples. Fig.1b and 1c show a two-dimensional AFM and phase images of Ni/Cu multilayer. These films had an average roughness of 20–30 nm. Fig.2 shows a 3-dimensional AFM image of Ni/Cu/Ni/Cu/Ni multilayer.

The X-ray diffraction patterns of the typical Cu/Ni multilayers are shown in Fig.3. From the X-ray diffraction pattern, a strong (111) reflection of the Ni/Cu multilayers was found to occur between the (111) Bragg peaks of Cu and Ni, which indicates the presence of a single phase structure. It can be seen in the 2θ range of 40–45°. The XRD data show two different diffraction peaks, corresponding to the (111) crystallographic plane of Ni (d_{Ni}=2.0403 Å) and the (111) plane of Cu (d_{Cu}=2.034 Å). There are also seen the same peaks around (200). The peaks around (111) reflection are stronger in intensity than (200) peaks. The X-ray analysis revealed that inside the applied angle range the (111) and (200) reflexes arising from Cu and Ni, respectively, were present. The proximity of diffraction peaks (111) for Cu and Ni, and (200) for Cu and Ni did not allows one to separate these peaks under measurement conditions.

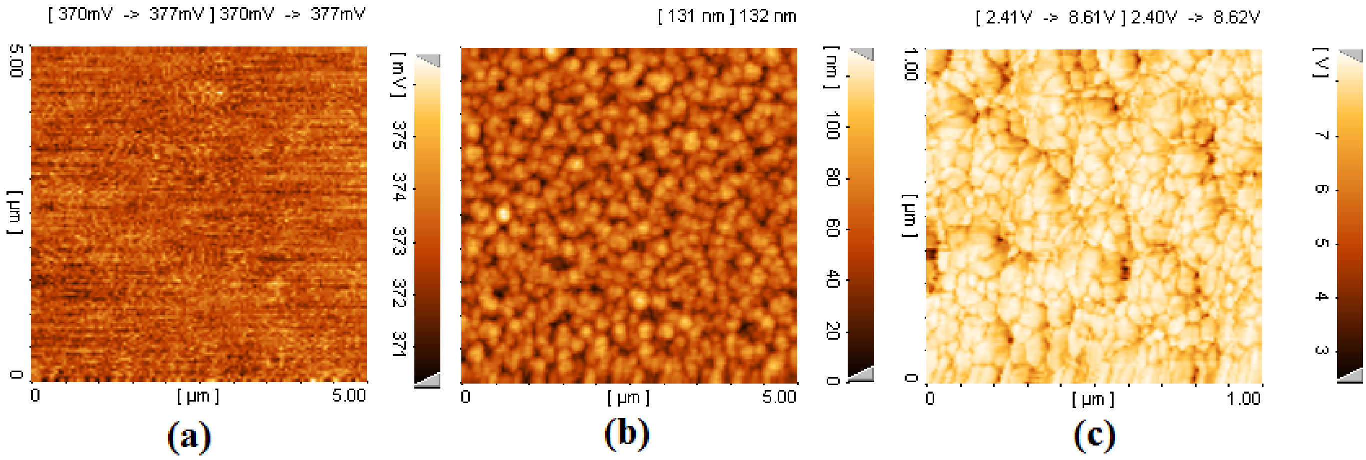

Fig.1. (a), (b) and (c) are MFM, AFM and phase images of Ni/Cu/Ni/Cu/Ni, respectively.

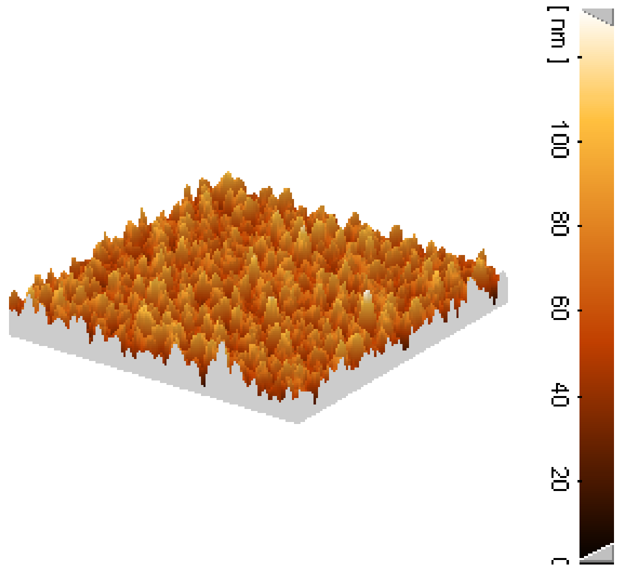

Fig.2. 3-dimensional topographical picture of Ni/Cu/Ni/Cu/Ni multilayer.

The specimens were then subjected to isothermal heat treatment for the purpose of annealing them in a ~10^{-5} mbar vacuum. The specimens were heated in a HT-XRD chamber from room temperature to 625°C for a period of 2 hours. Figure 4 shows the result. The wide angle diffraction patterns Fig. 5 indicated that a predominantly (111) texture was preserved during annealing. This indicates that for Ni/Cu multilayer coatings deposited by RF(DC) magnetron sputtering, the substrate temperature plays an important role in the formation of superlattice structure. The analysis of the inter-planar distances connected with the (200) reflexes does not indicate their distinct increase with temperature. Up to 300°C, the d_{200} values decrease, and only after heating at 350°C they increase up to the theoretical value. For all multilayers, the intervals between points representing d values for 250 and 350°C have a nearly identical slope.

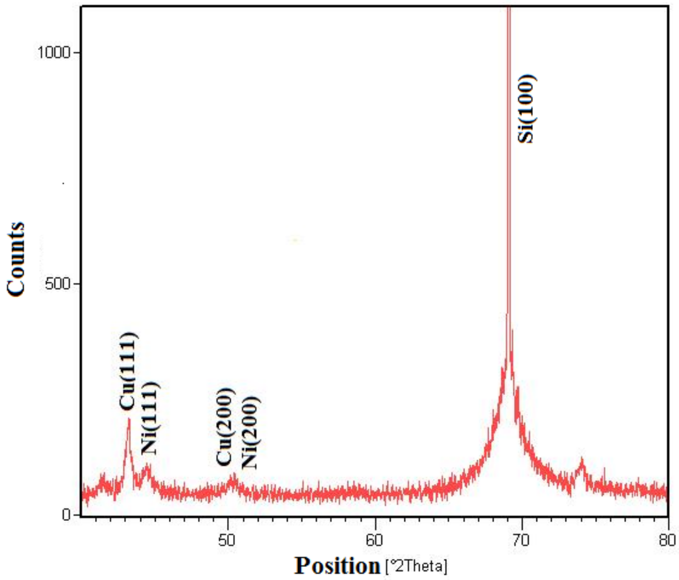

Fig. 3. X-ray diffraction patterns of Ni/Cu multilayer coatings deposited on Si(100) substrates at 25°C.

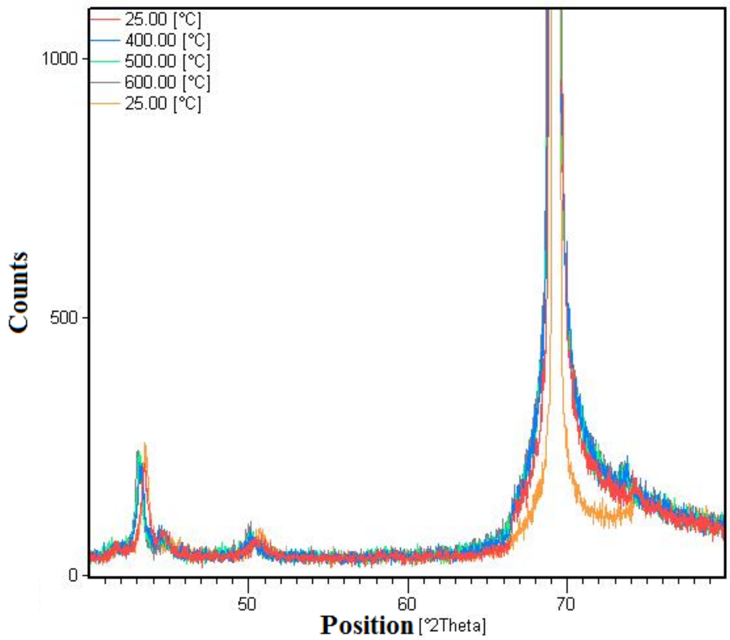

Fig.4. X-ray diffraction patterns of Ni/Cu multilayer coatings deposited on Si(100) substrates annealed up to 625°C.

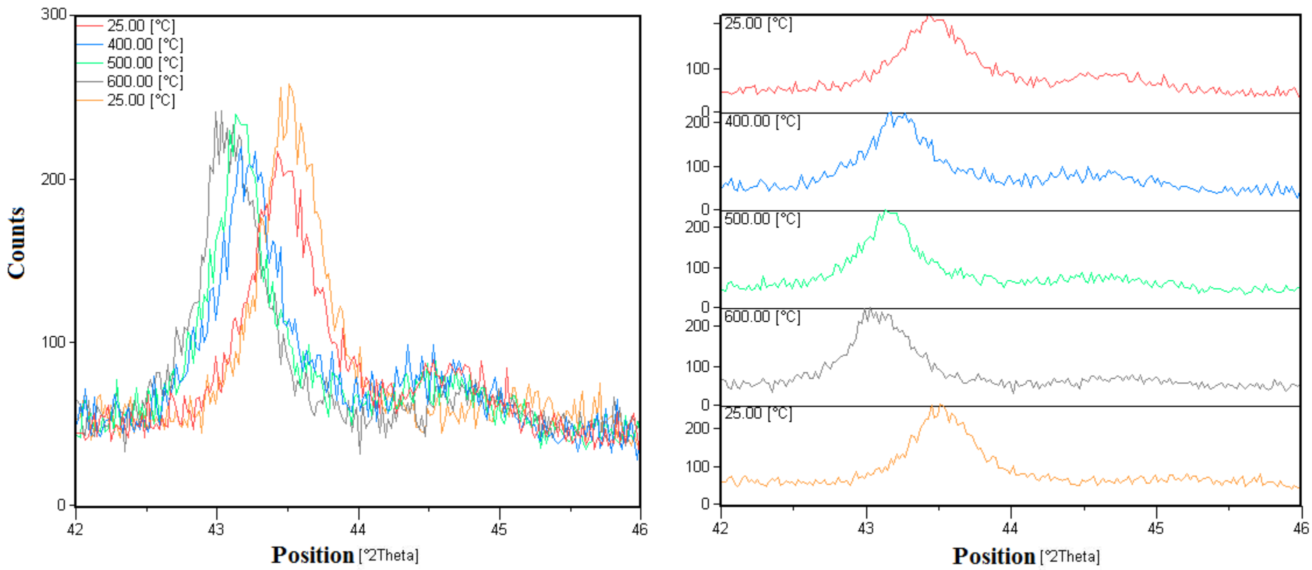

Fig.5. Wide angle X-ray diffraction patterns of Ni/Cu multilayer between 42-46°.

4. Conclusions

Ni/Cu multilayer coatings were deposited using an RF(DC) magnetron sputtering process. Films showed the appearance of peaks along the (111) and (200) principal reflections in the XRD data, indicating the formation of a superlattice structure. The HT-XRD results also show that the substrate temperature plays an important role in the formation of superlattice structure. The XRD patterns of the annealed samples showed that if the temperature and time of annealing increase, the satellite peaks begin to disappear. It means by increasing these two parameters, the sharpness of the bilayer interface decreases and the multilayered structure tends to become alloy structure.

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