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Study of Magnetic Properties and Characterization of Ni/Cu Multi-Layer Structures Using in-situ High-Temperature X-Ray Diffraction (HT-XRD)

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Abstract

In this paper, the results of magnetic and structural measurements of Ni/Cu multi-layers with different thicknesses of Cu and Ni grown on Si (100) substrate are presented. The Ni/Cu multi-layers nanostructure is analyzed using X-ray diffraction (XRD) diagram at room temperature and X-ray diffraction in a vacuum at high temperature (HT-XRD). The periodic peaks in the XRD spectrum, the multilayer structure and thus, the superlattice structure of the films are confirmed. But the in-situ XRD spectrums at high temperature reveal that the increase of temperature and annealing time is associated with the decrease or disappearance of intensity of the multilayer peaks and periodic peaks. It means the increase of these two parameters is accompanied with the weakening of interface sharpness between the layers, and the samples' multilayer structures are inclined toward the alloy structure.

Keywords: Electro deposition; Ni/Cu multi-layer; very thin film; in-situ X-ray diffraction at high temperature (HT-XRD)

Introduction

Multi-layer thin metallic systems are often used in reading the heads of magnetic disk and magnetic field sensors as well as in magnetic recorders. To achieve a higher density for data packets, the boundaries of multi-layers are included in the total volume of multi-layer system, thus, the physical state of the interface has a significant impact on the electrical, magnetic and structural properties of the system as a whole. Recently, nanostructure materials such as modulated alloys in the form of composites and multi-layers have been studied extensively.

The main objective of such studies is to investigate the magnetic and electrical properties (GMR effect) and surface topography (Anderson et al., 2003, Baibich et al., 1988). The mechanical properties of these surfaces have also been studied extensively (Chassaing, 1997; Cziraki, 2003; Haseeb et al., 2003; Liu et al., 2005). Despite the limitations such as the conductivity of the substrate, in the past decades some electrochemical methods have been used for the preparation of multi-layers like Ni/Cu (Ludwig et al., 2003), NiCO/Cu (Georgescu and Georgescu, 2002), CO/Cu (Gomez et. al., 2005), NiFE/Cu (Nabiyouni and Schwarzacher, 2005) and CO/PT (Zhang et. al., 2004).

In this paper, the growth of Ni/Cu multi-layers on silicon wafers is reported using magnetron sputtering method. The polarization of Si (100) substrates with respect to the potential deposition effects of substrates on Cu and Ni is studied. Characterization of multi-layer nanostructure with high-angle X-ray diffraction (XRD) and X-ray diffraction at high temperatures in vacuum (HT-XRD) are presented. It is also shown how structural characteristics depend on the thickness of all the deposited layers. The paper aims at studying Ni/Cu multi-layer grown on Si (100) substrate with vacuum heating in situ. At the end, the effect of temperature on the magnetic properties of nickel and copper bilayers are discussed.

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Materials and methods

Alternating layers of copper and nickel with different thicknesses were prepared on substrates made of silicon at different temperatures using RF magnetron sputtering method. Single-crystal Si wafers with preferential orientation (100) were used as the substrate for deposition. Before placing the samples in a vacuum chamber to perform H-terminate, the substrate surface was placed in HF (% 10) solution for one minute, and then, the standard methods used in microelectronic technology were employed for cleaning the samples. The Ni and Cu targets had a purity of 99.94% or more and deposition on the substrate was undertaken with the mass flow rate of 20.4 cm3min-1. The deposition of Ni/Cu multilayer occurred with base pressure of 10-6 torr and argon gas pressure of 60 mbar; the distance between the substrate and the target was 0.054m. In these experiments, the rates of deposition of 1 and 1.6 were respectively used for the Cu and Ni targets; the layers thickness was set using a quartz crystal thickness gauge. The crystal structure and phase composition of thin films were determined by X-ray Diffraction device (PHILIPS-X'Pert) and Cu-Kα Radiation (λ = 1.5406Å). In this paper, the surface morphology was studied employing atomic force microscopy device (AFM) and magnetic properties of the samples were evaluated using MFM method.

Experimental results and discussions

Magnetic fields and topography of the samples surfaces (MFM and AFM) are shown in Figures 1 and 2. In a non-contact technique, the MFM device has a dual-pass action with tips coated by vertical magnetized CoCr. According to the captured images, the distance from the tip to the sample surface or the scanned height is 5μm which is shown in Figure 1. In the images, the bright areas (dark) are corresponded to status of low (high) areas. The alternate period or the fields' width depends on the radiation dose with a range between 2 and 4 micro meters. Measurements undertaken on different magnetic samples indicate that the distance between the needle tip and the effective position of magnetic moment in the tip depends on the magnetic properties of the measured surface; thus, the magnetic moment calculated from the MFM data should be considered as a rough estimate.

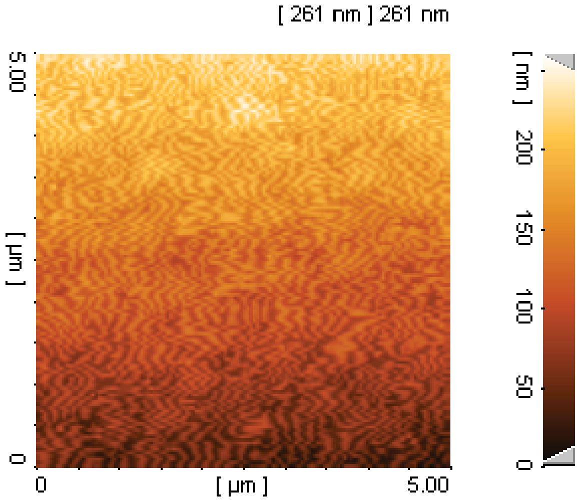

Figure 1. Three-dimensional MFM image of the five-layered Ni–Cu

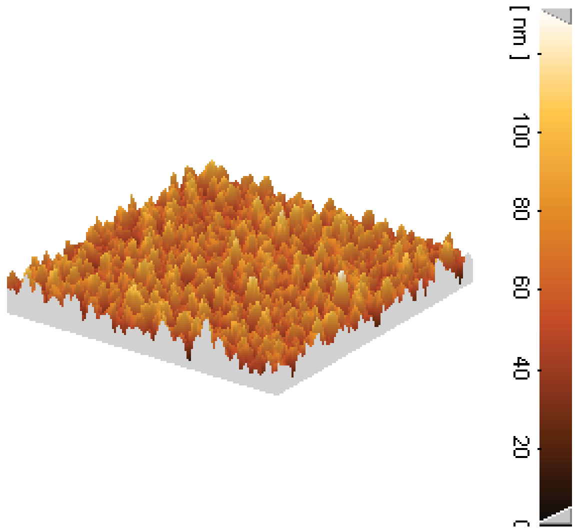

Figure 2. Three-dimensional AFM image of the five-layered Ni–Cu

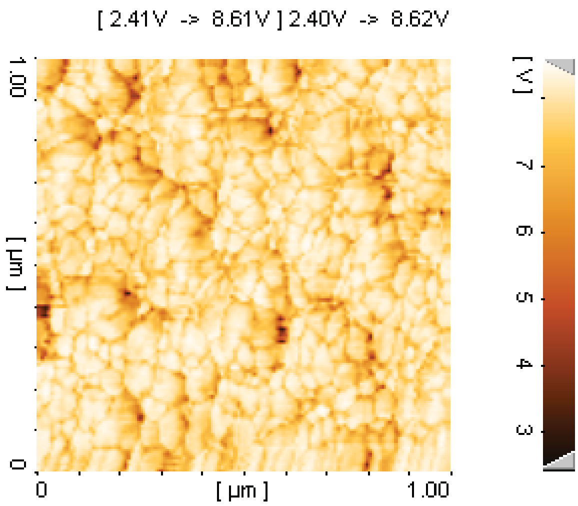

Figure 3. Phase image of the five-layered Ni–Cu

Topographic characteristics of the surface provide some significant information about the samples. Figs. 2 and 3 show the three-dimensional AFM and phase images of Ni/Cu multilayer. In these layers, a medium roughness about 20 to 30 nm is observed. Fig. 2 shows the three-dimensional AFM image of a five-layered Ni/Cu.

In the X-ray diffraction spectra of all samples, the main peak and the periodic peak are observed. The main peak is corresponded to the successive atomic plates of multilayer components and the periodic peaks are corresponded to the periodic structure of paired layers. The existence of periodic peaks confirms the superlattice structure of the layers.

Fig. 4 shows the main peaks of Cu (111) and Cu (200) in the substrate face center cubic Brag structure (fcc). The two main peaks relate to the multilayer structure plates (111) and (200) such that their angular positions correspond to the average distance between the constitutive atoms of multilayer.

Based on the X-ray diffraction patterns, a strong peak (111) of Ni/Cu multi-layer occurs between the Brag peaks (111), nickel and copper - which indicates the presence of an individual phase structure; it is observable in 2θ in the range of 40-45 degrees. According to the XRD information in relation to two different peaks corresponding to (111) d-Spacing nickel and copper plates, (dNi =2.0403 E) and (dCu =2.034 E) are obtained, respectively. In addition, similar peaks are seen around (200). The peaks around reflection (111) are stronger than (200) in terms of intensity. To check the temperature changes of the samples structure at high temperatures, they are exposed to the heat treatment in a vacuum chamber with 10-4 Mbar pressure. During the temperature increase, the XRD spectra of the samples are obtained at the room temperature at the beginning and at the end as well as at 400, 500 and 600 ° C (a two-hour period).

As it can be seen from the diagram shown in Fig.5, there are significant changes in the peak positioned in 69°. With the increase of temperature, its height is lowered and in the 25° in backward path it is about one-twentieth of 25° in forward path i.e. after annealing, the peaks are wider and crystalline sizes are smaller. Fig. 6 shows diffraction patterns with wide-angle ranges indicating the dominance of structure (111) during annealing. This indicates that for the deposition of Ni/Cu multi-layer by RF magnetron sputtering, temperature plays an important role in the structure of the superlattice; but the inter-plate distance in relation to individual reflections (200) with increasing temperature is not obvious.

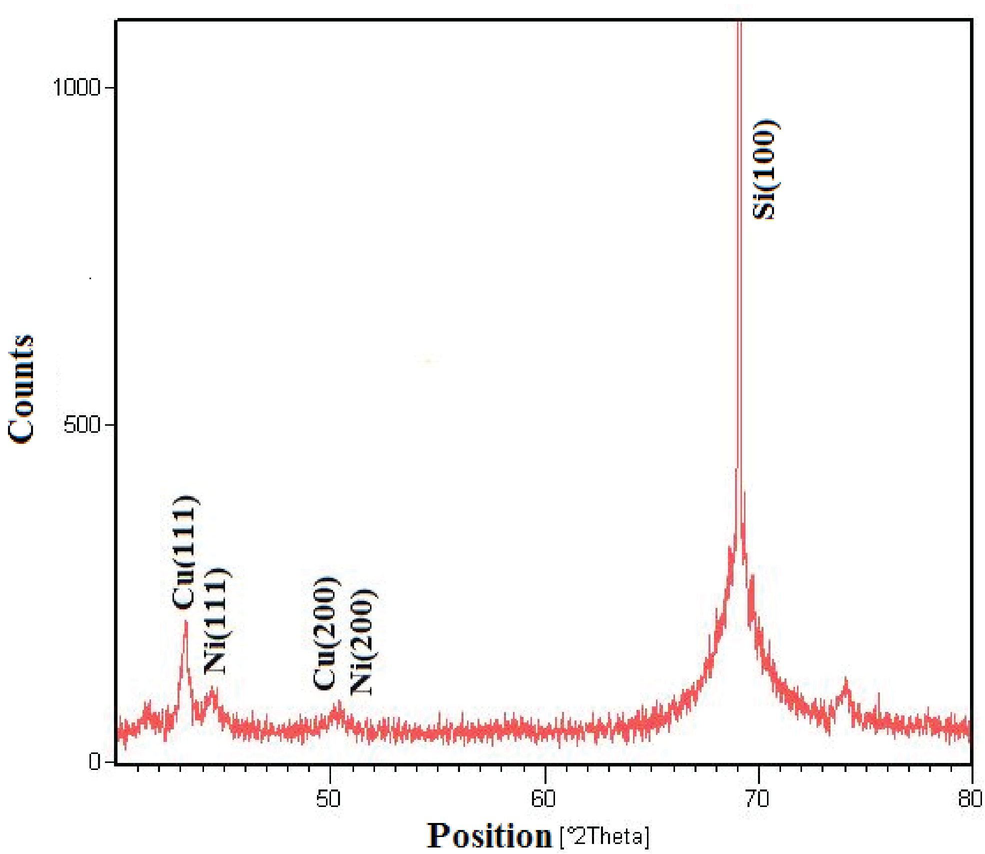

Figure 4. The XRD diffraction pattern of the nickel - copper multi-layer

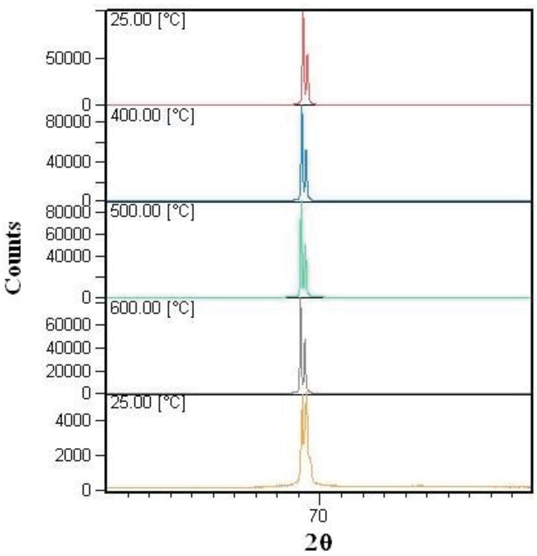

Figure 5. Peak changes at different temperatures

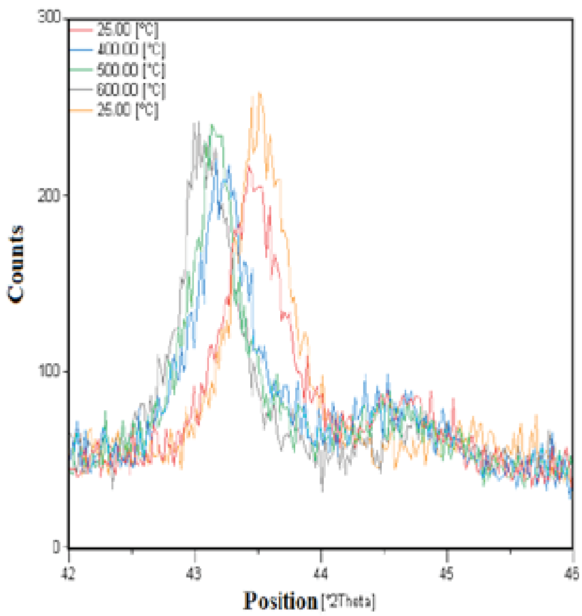

Figure 6. Peak movement at different temperatures

As it can be seen from Fig.6, the increase of temperature is associated with decrease of d200 value in (200), while it increases to maximum at 600°. At the room temperature, it returns to the previous value which means the samples structures are reversible.

As it was mentioned previously, the comparison of curves in Fig.6 reveals that the increase of multilayer annealing temperature and time is associated with the gradual disappearance of periodic peaks and also multilayer peaks (111) and (200); in this way, the alloy structure is substituted with multilayer structure. It is because at the higher temperature, atoms move faster and so nickel atoms are distributed in the copper layer and copper atoms are distributed in the nickel layer. As a consequence, the uniformity of the interface between the mentioned layers is reduced and the multilayer structure tends to the alloy structure.

Conclusions

The Ni/Cu multilayer is deposited using RF magnetron sputtering process. The layers peaks in the main reflections (111) and (200) are shown in the XRD data. The information indicates that it is a superlattice structure. The results of HT-XRD also reveal that the substrate temperature play an important role in the information of the superlattice structure. The post-annealing XRD spectrum of samples shows that the increase of annealing temperature and time is associated with the decrease or even disappearance of peaks related to multilayer and periodic peaks. It means that the increase of these two parameters is accompanied with the weakening of interface sharpness between the layers, and the samples' multilayer structures are inclined toward the alloy structure.

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