Surface Analysis of Impact Compressed Cu-Ni Multilayer

T. Une, J. W. Lee¹, M. Nakamura², and T. Tanaka*

Hiroshima Institute of Technology, Saiki-ku, Hiroshima 731-5193

¹Dept. of Physics, Hallym University, ChunChon, KangWon-Do, 200-702, Korea

²Fujitsu Analysis Laboratory LTD, 4-1-1, Kamikodanaka, Nakahara-ku, Kawasaki

211-8588

(*tanaka@cc.it-hiroshima.ac.jp)

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Surface analytical investigation with AES and XPS has been performed on Cu-Ni multilayer and its impact compressed sample. A well formed Cu-Ni multilayer using RF magnetron sputtering system with MMPC has been observed. The possibility of the formation of a Cu-Ni alloy after the high speed impact compression has been found, and this suggests that the impact compression method is useful for the preparation of metal alloys.

1. INTRODUCTION

Recently, the occurrence of giant magnetoresistance (GMR) and its dependence on both the magnetic and non-magnetic layer have been observed in the electrodeposited Cu-Ni/Cu multilayers [1]. However, it is difficult to make such Cu-Ni multilayers because of the easy diffusion property of the substances. In this paper, we report experimental results on Cu-Ni multilayers deposited by RF magnetron sputtering system with Multipolar Magnetic Plasma Confinement (MMPC) [2] and the formation of Cu-Ni alloys by using high speed compression applied on the Cu-Ni multilayers. Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) with depth profiling analysis have been performed for this study.

2. EXPERIMENTS

2-1 Fabrication of Cu-Ni multilayers

The deposition of Ni and Cu thin films on

the Ni substrate (99.999%) was alternatively deposited using the RF magnetron sputtering system with MMPC. A schematically drawn diagram of the system is illustrated in Fig. 1. The MMPC has been employed for the Ni deposition whereas it was not the case for the Cu deposition. The magnetron discharge was generated by RF power (13.56 MHz).

Fig.1 Schematic diagram of the RF magnetron sputtering system with MMPC

The sputtering gas was Ar with the gas flow rate of 10 sccm. The substrate temperature was kept at room temperature. The RF power was 100 W for Cu and 150 W for Ni. Five alternative layers with the whole thickness of about 160 nm have been deposited on the Ni substrate which is shown in Fig. 2.

Fig.2 Schematically drawn cross section of the Cu-Ni multilayer sample

2-2 High speed impact compression

High speed compression on the Cu-Ni multilayer samples has been performed at the ultra-high-speed impact loading machine installed at the research center of Hiroshima Institute of Technology. Bullet mass of 40 g and bullet speed of 30 m/s have been used as the compression parameters for the impact experiment. Fig.3 shows the impact compression situation at the loading machine.

Fig.3 High speed compression on the sample at the loading machine.

2-3 Surface characterization

The Cu-Ni multilayer samples and the impact compressed samples have been characterized by using conventional AES and XPS apparatus. The experimental conditions for the surface analysis and depth profiling are summarized in Tables 1 and 2.

Table 1 Experimental conditions for AES analysis

Probe Energy	10 kV
Probe Current	about 15 nA
Probe Angle of Incidence	60°
Ion Energy	1 kV
Ion Angle of Incidence	about 58°
Ion Etching Method	Zalar Rotation
Analysis Area	2 μm $\times$ 2.3 μm (as deposition)
	10 μm $\times$ 11.5 μm (after high speed compression)

Table 2 Experimental conditions for XPS analysis

X-ray Source	MgK α
X-ray Source Power	8 kV
	30 mA
Ar ⁺ Etching Power	1.5 kV
	20 mA
Ion Beam Angle of Incidence	30°
Ion Etching Method	Raster Scan
Analysis Area	5mm $\times$ 5mm

3.RESULTS AND DISCUSSION

Figures 4,5 and 6,7 show the AES and XPS sputter depth profiles of Cu-Ni multilayers before and after the high speed compression, respectively.

3-1 Cu-Ni multilayer samples

Figure 4 shows the AES sputter depth profile of the five layers of a Cu-Ni multilayer with atomically flat interfaces. For quantitative investigation of the interface, depth resolution of the interfaces has been calculated. Depth resolution is the depth range over which a signal increases (or decreases) by a specified amount when profiling through an ideally sharp interface between two media. By convention, the depth resolution corresponds to the distance over which a 16% to 84% (or 84% to 16%) change in signal is measured [3]. The calculated depth resolution of the five interfaces is summarized in Table 3.

Table 3 Calculated depth resolution with AES
Cu (920 eV) and Ni (848 eV) signals

Interface	1st	2nd	3rd	4th	5th
Cu signal	4.6nm	4.9nm	7.7nm	7.2nm	8.0nm
Ni signal	4.9nm	4.6nm	7.6nm	6.9nm	7.8nm

The depth resolution has been increased from the first to the last fifth interface in spite of the usage of the Zalar rotation. This may come from the increase of the surface roughness during the sputtering.

Table 4 Calculated depth resolution with XPS
Cu signal

Interface	1st	2nd
Cu signal	9.6nm	10.6nm

Fig. 4 AES sputter depth profile of Cu-Ni multilayer

Fig. 5 AES sputter depth profile of the multilayer after high speed

Fig. 6 XPS sputter depth profile of Cu-Ni multilayer

Fig. 7 XPS sputter depth profile of the multilayer after high speed

The depth resolution of the first and second interfaces with XPS measurement has been calculated and summarized in Table 4. It is given on this table that the depth resolution of second interface is 10.6 nm by calculating the 84% to 16% distance of Cu signal in Fig.6 which shows a XPS sputter depth profile. This may come from the increase of the surface roughness due to the large analysis area of the XPS rather than that of the AES.

3-2 Impact compressed samples

A Cu-Ni alloy layer is formed at the depth range from 20 nm to 65 nm below the surface by the high speed impact compression method based on the AES depth profile shown in Fig.5. But two cases are possible to give rise to a mixed profile. One case is the mixing between Cu layer and Ni layer each other on the viewpoint of microstructure, but Cu- and Ni-phases exist separately. Another case is the alloying phenomenon with the formation of a Cu_{0.5}Ni_{0.5} phase. But further investigation using Transmission electron microscopy (TEM) is needed in order to verify the existence of an alloy phase.

4.CONCLUSIONS

Cu-Ni multilayer samples and impact compressed samples have been characterized with AES and XPS depth profiling. A well formed Cu-Ni multilayer using RF magnetron sputtering system with MMPC has been observed. The possibility of the formation of a Cu-Ni alloy after the high speed impact compression has been found. Thus, it has been demonstrated that the high speed compression may become one of a useful method for the preparation of metal alloys.

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