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Preparation of Smooth Self-Supporting Ni/Cu Multilayer

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[Ni(5 nm)/Cu(5 nm)]$_{50}$ multilayer was deposited on SiN membranes by DC magnetron sputtering. Field emission scanning-electron microscopy (FESEM), atomic-force microscopy (AFM), and X-ray diffraction (XRD) were used to analyze the surface morphology and the microstructure. The surface of the multilayer is continuous and flat, with root-mean-square roughness (RMS) of 9.9 nm. The period of the multilayer is 10.7 nm, which is very close to the designed period of 10 nm. The multilayer was composed of multi-crystal. These results show that this experiment is a promising way to obtain flat self-supporting metal multilayer.

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I. INTRODUCTION

Self-supporting films play crucial roles in heavy ion accelerators, superconducting linear accelerators, high resolution time-of-flight (TOF) spectrometers, and other atomic- and nuclear-physics exploratory systems such as inertial-confinement fusion (ICF) experimental systems. Many self-supporting films have been successfully obtained in previous research. P. Maier-Komor *et al.* prepared self-supporting C films by using a laser plasma ablation deposition system [1–3]. Self-supporting W films [4], W-C films [5], Cr films [6], Be films [7], and large area C films with MgO coatings [8] were obtained by electron-beam vapor deposition. A modified *in-situ* polymerization method was used to obtain self-supporting polyimide films [9]. Liechtenstein *et al.* have prepared and investigated ultra-thin (1 μg/cm^2) diamond-like-carbon (DLC) foils by sputtering [10–12]. Self-supporting B films could be synthesized successfully by using electron bombardment and focused-ion sputtering [13]. To obtain self-supporting films, the film must grow on soluble substrates (*e.g.*, NaCl substrates) or parting agents. Restricted by the polishing treatment of single-crystal NaCl substrates, the roughness of the film is about 20 nm. Crystallized parting agents such as betaine + sucrose [14] and potassium oleate make the films deposited on them very rough. Soft parting agents (*e.g.*, potassium oleate+sucrose) will result in stress relief patterns on the Ni film. The mean roughness of these films is higher than 100 nm [15]. The difficulty in preparing self-supporting multilayers with a flat surface and interface has restricted their application.

This paper provides a process to fabricate self-supporting Ni/Cu multilayer with low roughness.

II. EXPERIMENTS AND DISCUSSION

SiN film with thickness of 1 μm was prepared by plasma enhanced chemical vapor deposition (PECVD) on both sides of Si wafers. A window pattern was formed on the SiN layer of the back surface. The SiN layer of the front sidurface was used as an etch stopper. Next, the Si substrate in the window area was chemically etched off with KOH to yield a free-standing SiN membrane. The SiN membrane is shown in Fig. 1.

Ni/Cu multilayer was deposited on the SiN membranes by DC magnetron sputtering at room temperature. The nominal thickness of the Ni and the Cu layer was 5 nm, respectively. There were 50 pairs in the Ni/Cu multilayer. The base pressure was lower than 1×10^{-4} Pa, and the Ar pressure during the sputtering was 1 Pa. The

Fig. 1. Photograph of SiN membrane.

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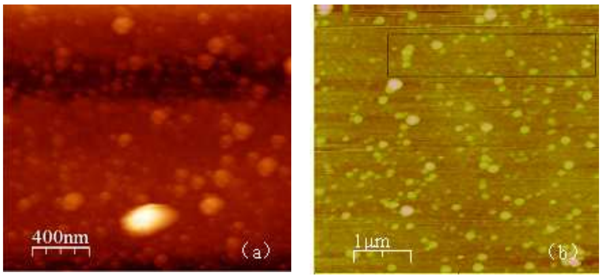

Fig. 2. (Color online) AFM images of Ni/Cu multilayer and SiN membrane: (a) image of the Ni/Cu multilayer, gained by AFM Pico Plus; (b) image of the SiN membrane, gained by AFM NanoIIIa.

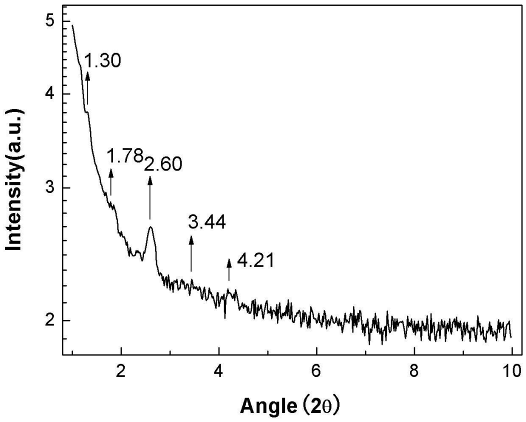

Fig. 3. Low angle XRD pattern of Ni/Cu multilayer.

deposition rate for Ni and Cu was 0.09 nm/sec and 0.08 nm/sec, respectively.

The morphologies of the multilayer and the SiN membrane were analyzed by using an AFM Pico Plus and AFM NanoIIIa. The modulation period Λ of the multilayer was investigated by low-angle X-ray diffraction by using a Rigaku D/max-RB with Cu K_α radiation. Structural investigations were carried out by grazing incidence x-ray diffraction (XRD) by using an X'Pert Pro MPD.

A 2×2 μm^2 AFM image of the Ni/Cu multilayer is displayed in Fig. 2(a). Round shaped particles with average diameter about 150 nm can be seen on the surface. The RMS roughness of the multilayer is 9.9 nm. Some particles with average diameter about 200 nm can also be seen on the surface of the SiN membranes (Fig. 2(b)).The RMS roughness of the SiN membranes is 2.9 nm. We speculate that the Ni/Cu multilayer replicates the surface features of the SiN membranes, which results in large particles in the multilayer, surface roughness, and interfacial roughness. However, the roughness of the multilayer is much lower than that of the self-supporting films made by other methods [15].

The low-angle XRD peaks induced by the compositional modulated structure of the Ni/Cu multilayer are shown in Fig. 3. The modulation period for the multilayer was calculated from the Bragg equation, modified

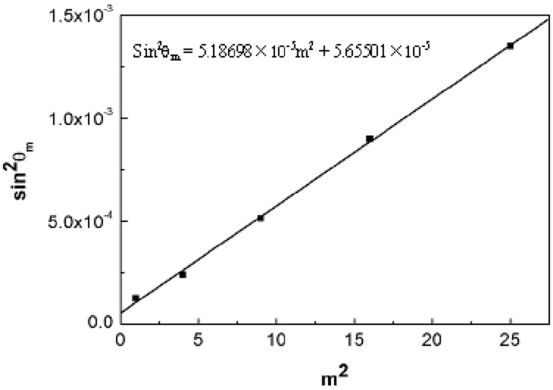

Fig. 4. Plot of $\sin^2\theta_m$ *vs.* m^2 with fitted curve.

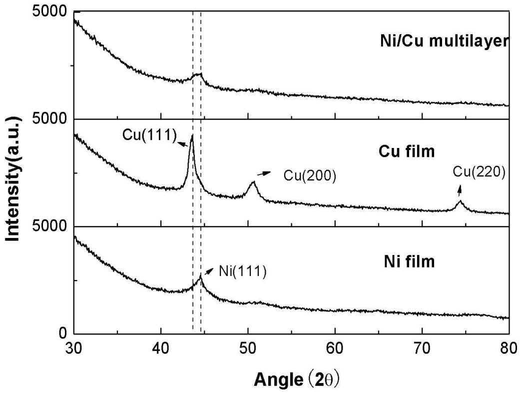

Fig. 5. Grazing incidence XRD patterns of Ni/Cu multilayer, Cu film and Ni film.

for refraction [16]:

$$\sin^2(\theta_m) = (\frac{m\lambda}{2\Lambda})^2 + 2\delta, \quad (1)$$

where Λ is the modulation period, m the integer reflection orders number, and δ the average deviation of the refractive index from unity. By using Eq. (1), Λ and δ can be determined from the linear regression of $\sin^2\theta_m$ *vs.* m^2. Figure 4 shows the plot of $\sin^2\theta_m$ *vs.* m^2. As expected from Eq. (1), all the data points fit very well on a straight line, from which Λ and δ are determined. The value of Λ is 10.7 nm, which is very close to the designed period of 10 nm.

The peaks can be recognized in Fig. 3, but their intensity is very weak. Two reasons lead to this. Cu and Ni are mutually soluble in each other in the solid state for all compositions, so the interdiffusion between the layers is strong. Another factor that results in weak peaks is the roughness of the SiN membrane, which is shown in Fig. 2(b). This replicated roughness from the SiN membrane induces non-parallel interfaces.

Grazing incidence XRD is a well-established technique to determine the crystal structure of films. Figure 5 shows the XRD pattern of the Ni/Cu multilayer. The patterns of Ni film (100 nm) and Cu film (100 nm) made in the same condition are also shown in Fig. 5. The microstructures of the monolayers and the multilayer are all polycrystalline. The peak of the multilayer locates between the Ni(111) peak and the Cu(111) peak. These results suggest the Cu-Ni solid-solution alloy is formed on the interface of the multilayer. The peak of the multilayer is lower and wider than those of the monolayers, although the total thickness of the multilayer is larger. This reveals that the crystalline grains of the multilayer are smaller than these of the monolayers .

III. CONCLUSIONS

A self-supporting Ni/Cu multilayer with roughness lower than 10 nm can be made on SiN membranes by DC magnetron sputtering. This roughness is much lower than that of self-supporting films made by other methods. The multilayer period is 10.7 nm, which is very close to the designed period of 10 nm. The structure of the multilayer is polycrystalline and Cu-Ni solid solution alloy is formed on the interface of the multilayer.

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REFERENCES

[1] P. Maier-Komor, G. Dollinger and R. Krücken, Nucl. Instr. Meth. A **56**, 14 (2006).
[2] P. Maier-Komor, G. Dollinger and H. J. Kürner, Nucl. Instr. Meth. A **438**, 73 (1999).
[3] P. Maier-Komor, G. Dollinger, C. M. Frey and H. J. Kürner, Nucl. Instr. Meth. A **362**, 208 (1995).
[4] J. O. Stoner, Jr. and C. S. Jolivet, Nucl. Instr. Meth. A **561**, 11 (2006).
[5] J. O. Stoner, Jr., Nucl. Instr. Meth. A **521**, 43 (2004).
[6] J. O. Stoner, Jr., Nucl. Instr. Meth. A **480**, 44 (2002).
[7] J. O. Stoner, Jr., Nucl. Instr. Meth. A **397**, 68 (1997).
[8] A. Stolarz and P. Maier-Komor, Nucl. Instr. Meth. A **480**, 194 (2002).
[9] A. Stolarz and J. Van Gestel, Nucl. Instr. Meth. A **561**, 115 (2006).
[10] V. Kh. Liechtenstein, T. M. Ivkova, E. D. Olshanski, R. Repnow, P. Steier, W. Kutschera, A. Wallner and R. von Hahn, Nucl. Instr. Meth. A **561**, 120 (2006).
[11] V. Kh. Liechtenstein, T. M. Ivkova, E. D. Olshanski, R. Golser, W. Kutschera, P. Steier, C. Vockenhuber, R. Repnow, R. von Hahn, M. Friedrich and U. Kreissig, Nucl. Instr. Meth. A **521**, 197 (2004).
[12] V. Kh. Liechtenstein, T. M. Ivkova, E. D. Olshanski, I. Feigenbaum, R. DiNardo, M. Döbeli, A. M. Baranov, R. Repnow, R. Hellborg, R. A. Weller and H. L. Wirth, Nucl. Instr. Meth. A **397**, 140 (1997).
[13] G. J. Xu and Y. Q. Wei, Nucl. Instr. Meth. A **397**, 62 (1997).
[14] P. Maier-Komor, Nucl. Instr. Meth. A **102**, 485 (1972).
[15] F. J. Gao, R. T. Zheng, G. A. Cheng and T. F. Yang, Nucl. Instr. Meth. A **577**, 397 (2007).
[16] B. K. Agarwal, *X-ray Spectroscopy* (Springer, Berlin, 1979), p. 134.