Available online at www.sciencedirect.com
ScienceDirect
Nuclear Instruments and Methods in Physics Research A 577 (2007) 397–401
NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH
Section A
www.elsevier.com/locate/nima

Influence of substrates on roughness of self-supporting Ni films

Fengju Gao, Ruiting Zheng*, Guoan Cheng, Tengfei Yang

Key Laboratory of Radiation Beam Technology and Material Modification of Education Ministry, Department of Materials Science & Engineering, Beijing Normal University, Beijing Radiation Center, Beijing 100875, PR China

Received 25 October 2006; received in revised form 27 March 2007; accepted 30 March 2007
Available online 18 April 2007

Abstract

Self-supporting Ni foils were obtained by floating of Ni films from water soluble substrates. Ni films were prepared by metal vapor vacuum arc (MEVVA) ion deposition. The following release agents were applied on Si wafer substrates: betaine monohydrate with sucrose, potassium oleate, potassium oleate with sucrose. In addition polished NaCl crystals and self-supporting collodion foils were used as soluble substrates, respectively. Field emission scanning electron microscopy (FESEM) and atomic force microscope (AFM) were employed to analyze the surface morphology and the roughness of the Ni films and the substrates. The results indicate that the mean roughness of the self-supporting Ni foils depends on their substrates. Self-supporting collodion foils seem to be one of the most suitable candidates for preparing self-supporting Ni foils with low roughness. Mean roughness of the best self-supporting Ni foils is about 3.8 nm.

PACS: 81.15.Ef; 29.25.−t; 68.55.Jk

Keywords: Release agent; Self-supporting target; Ni; MEVVA; Vacuum deposition

1. Introduction

Self-supporting foils play a crucial role in heavy-ion accelerators, super conducting linear accelerators, high-resolution time-of-flight (TOF) spectrometers and some other atom and nuclear physics explorations such as inertial confinement fusion (ICF) experiments. They can be used as stripper foils, secondary-electron emitters and targets. Due to selective transmission characteristic, certain self-supporting foils also have applications in the extreme ultraviolet and soft X-ray regions.

Many self-supporting foils had been successfully obtained in previous researches. P Maier-Komor et al.[1–3] prepared self-supporting C films with a total random orientation of nanocrystallites using a laser plasma ablation deposition system. Self-supporting tungsten foils [4], tungsten–carbon foils [5], chromium foils [6], beryllium foils [7] and large-area carbon foils with MgO coatings [8] were obtained by electron beam vapor deposition. A modified in situ polymerization method was used to get self-supporting polyimide foils with 25 cm diameter unsupported aperture [9]. Liechtenstein et al. [10–12] had investigated ultra-thin ($\leqslant 1\,\mu g/cm^2$) diamond-like carbon (DLC) foils prepared by sputtering. Self-supporting boron films could be prepared successfully using electron bombardment and focused ion beam sputtering [13]. Although the roughness of self-supporting foils affects their performance evidently, especially in the ICF and optical applications, few investigations had been carried out till now. Manufacturing of smooth self-supporting foils was still a great challenge.

In this paper, five different types of substrates were used for preparing self-supporting Ni foils. The influence of substrates on the surface morphology and roughness of Ni foils was investigated based on the experiments.

2. Experiment

2.1. Preparation of substrates

Five kinds of substrates were adopted for preparing the self-supporting Ni foils. They were Si wafer coated with betaine monohydrate + sucrose [14], Si wafers coated with

---

*Corresponding author. Tel./fax: +86 10 62205403.
E-mail address: rtzheng@bnu.edu.cn (R. Zheng).

0168-9002/$ - see front matter © 2007 Published by Elsevier B.V.
doi:10.1016/j.nima.2007.03.033

potassium oleate [15], Si wafers coated with potassium oleate + sucrose, self-supporting collodion foils and polished NaCl single-crystal substrates, respectively.

A de-ionized water solution of saturated betaine monohydrate + sucrose (7:1) was dropped onto a Si wafer and the wafer was rotated at 3000 rpm for 30 s. Then these substrates were dried at room temperature for more than 12 h. For the potassium oleate release agent coatings, substrates were prepared using saturated potassium oleate solution in alcohol. For the potassium oleate + sucrose substrate coatings, a mixture of potassium oleate with sucrose in water (mass ratio was 1:1:4) was applied. The procedure for coating these two substrate types were the same as described above for that of the betaine + sucrose coatings. To make a smooth self-supporting collodion film, collodion was dropped onto a Si wafer, which was rotated at 1000 rpm for 40 s. A frame with external dimensions of 2 cm × 2 cm and an aperture of 1 cm × 1 cm was glued onto the collodion, and then the frame with the collodion film was peeled from the Si wafer. The single-crystal NaCl substrates were obtained by silk fabrics polishing.

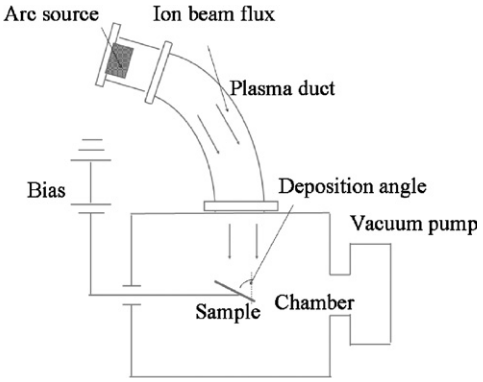

Fig. 1. Schematic diagram of a metal vapor vacuum arc (MEVVA) ion deposition system.

2.2. Deposition of the Ni films

The Ni films ($\approx 220\,\mu g/cm^2$ thickness) were deposited by a metal vapor vacuum arc (MEVVA) ion deposition system at ambient substrate temperature. Fig. 1 shows a schematic diagram of the facility. A Ni rod served as cathode for the Ni arc source. Ni plasma was generated by the arc between the cathode and the anode and fluxed into the chamber through a plasma duct in which large particles were filtered out. The remaining Ni ions were attracted by the bias voltage between the cathode and the substrate and thus deposited on the surface of the substrate. The ion beam flux was limited to 4 mA in the maximum during the deposition. The deposition rate ranged from 12 to $21\,ng\,cm^{-2}\,s^{-1}$. The high vacuum was better than 10^{-4} Pa.

2.3. Releasing of films and mounting of the foils

In order to get a self-supporting Ni foil, the Ni film must be removed from the substrate and mounted on a frame. For this, the film with which a Si wafer was coated had to be floated by dissolving the release agent film in a suitable liquid mostly in water. The substrates were gently lowered into a bath of deionized water at an angle of approximately 45°. After the parting agent layer had dissolved slowly, the Ni foil floated on the water surface. The water was carefully drawn off until the self-supporting Ni foil touched the target frame placed under it. Thus the foil was mounted on the frame.

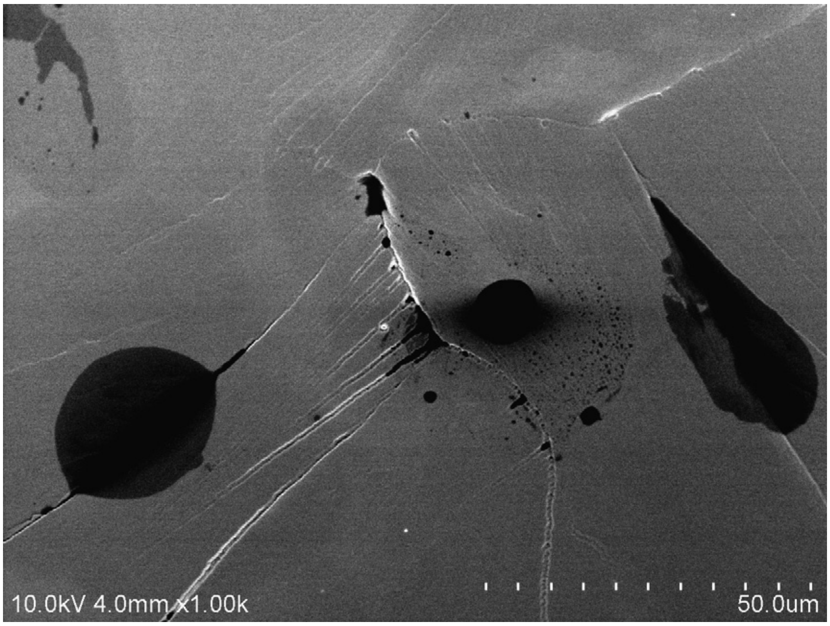

Fig. 2. SEM image of a $\approx 220\,\mu g/cm^2$ thick Ni foil floated from a betaine + sucrose parting agent.

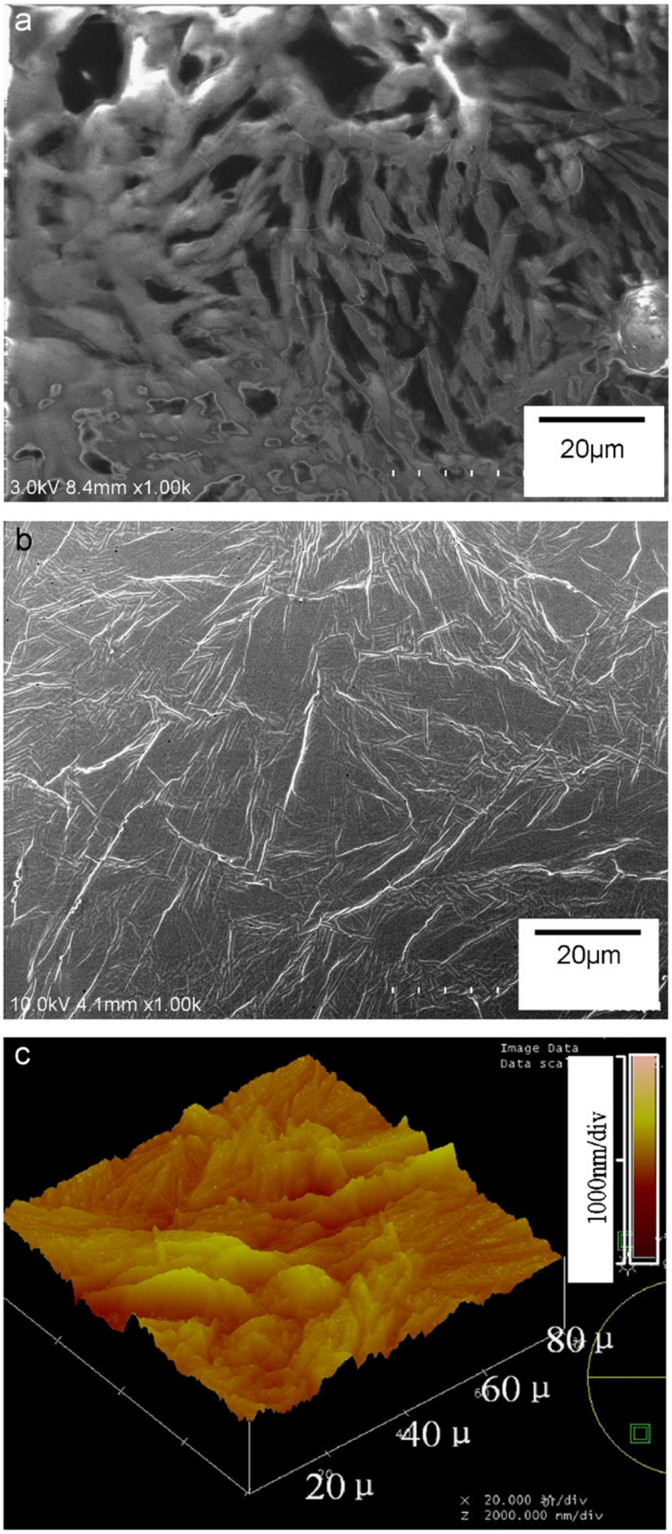

Fig. 3. Images of a potassium oleate film and of a Ni film deposited on potassium oleate on a Si substrate. (a) SEM image of a potassium oleate film. (b) SEM image of a ≈220 μg/cm^2 Ni film on potassium oleate. (c) AFM image of a ≈220 μg/cm^2 Ni film floated from potassium oleate.

The removal of the collodion was performed in acetone. The frame adhered with the Ni film and its supporting collodion film was submerged in acetone for about 5 min. When the collodion layer had dissolved, the Ni foil was gently picked up.

The morphology and roughness of the self-supporting Ni foils and the substrates were analyzed using a FESEM S-4800 (Hitachi High-Technologies Corporation, Japan) and an AFM NanoIIIa (Digital Instrument Company, US).

3. Results and discussion

The substrates show great effects on the surface morphology of thin Ni foils. Fig. 2 shows a SEM image of a Ni foil floated from the parting agent betaine monohydrate + sucrose. Some dendrite-like morphology and pores can be observed. Betaine monohydrate has a high solubility in water (160 g/100 g water). Betaine monohydrate + sucrose water solution as described above easily crystallizes in air, forming a dendrite-like morphology with interdendrite pores. The Ni foil replicates the surface feature of the release agent, which makes the Ni film deposited on it discontinuous and very rough.

A crystallized surface of a potassium oleate film can be seen in Fig. 3a. Dendritic-like morphology indicates that potassium oleate can also be easily crystallized. The Ni film also replicates the surface feature of the crystallized potassium oleate film (Fig. 3b). The dendrites of the potassium oleate are smaller than the ones of betaine + sucrose and their arrangement is more compact, so that the continuous Ni film is formed. This Ni foil with mean roughness (R_a) of 132.2 nm is smoother than the film deposited on the betaine + sucrose, as indicated by Fig. 3c.

To get smoother substrates, the crystallization of the parting agent must be suppressed or reduced. Sucrose could reduce the solubility of potassium oleate in water, and prevent further the crystallization of potassium oleate. Therefore the potassium oleate with sucrose solution is preferred as parting agent. Fig. 4a is a SEM image of a potassium oleate + sucrose film. As expected, the surface is rather smooth and almost no crystallization can be seen. But flower-like patterns are formed while the Ni film is deposited on the potassium oleate + sucrose (Fig. 4b). We speculate that the reason of this phenomenon comes from the inner stress. The hard Ni film deposited on a soft substrate forms wrinkles [16,17], which may come from the release of compressive stresses in the Ni film [18,19]. These patterns increase the roughness of the Ni foil. The relevant R_a reaches 145 nm.

NaCl single-crystal slices always have cleavage steps, which will affect further the roughness of the Ni film. So the NaCl substrate must be polished and be kept in an exsiccator filled with dry argon or it will deliquesce and become rough. The SEM and AFM images of Ni films deposited on polished NaCl substrates are showed in Fig. 5. Some scratch-like marks coming from the polished NaCl substrate can be seen on the surface of the Ni foil. R_a of the film is about 20 nm. Since NaCl is a kind of highly

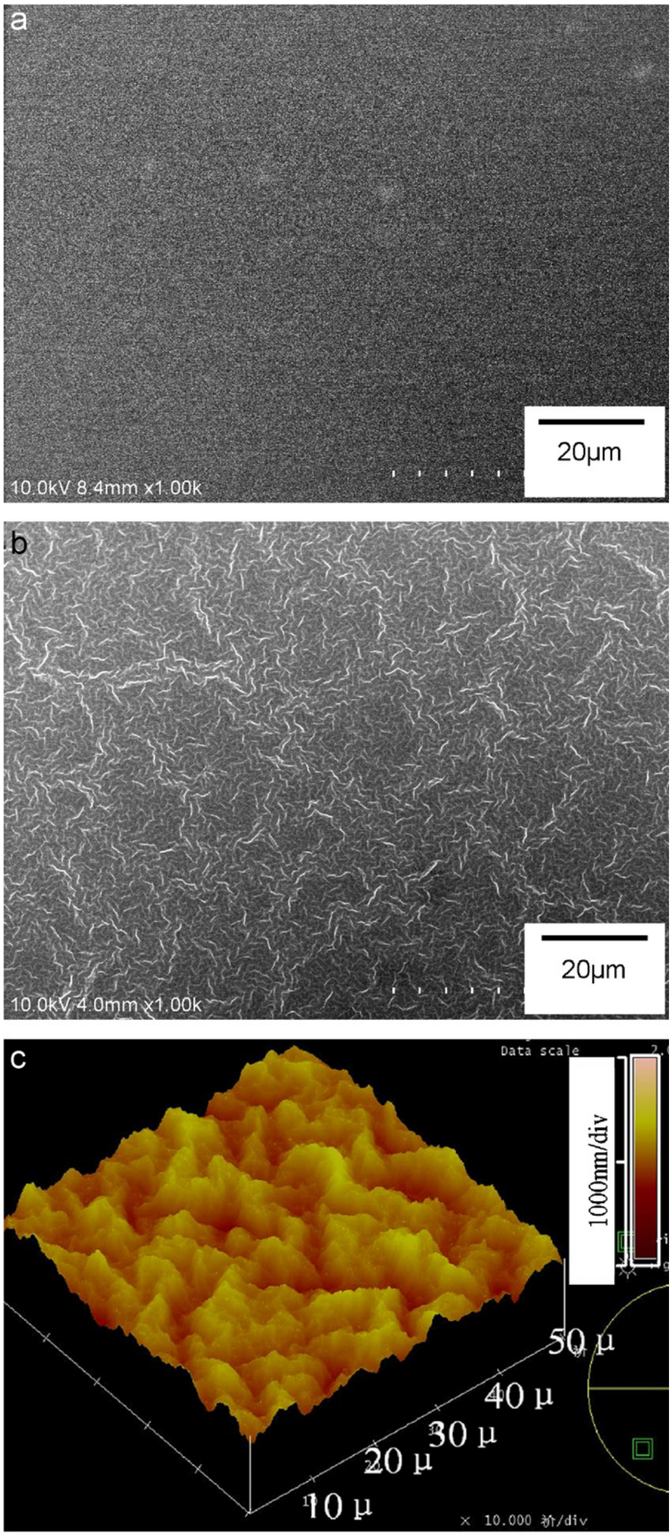

Fig. 4. Images of a potassium oleate + sucrose film and of a Ni film deposited on potassium oleate + sucrose on a Si substrate. (a) SEM image of a film of potassium oleate + sucrose. (b) SEM image of a ≈220 µg/cm^2 Ni film on potassium oleate + sucrose on a Si substrate. (c) AFM image of a ≈220 µg/cm^2 Ni film floated from potassium oleate + sucrose.

hygroscopic material, the Ni film must be removed from the NaCl substrate as soon as possible or it will break into small pieces rapidly in air.

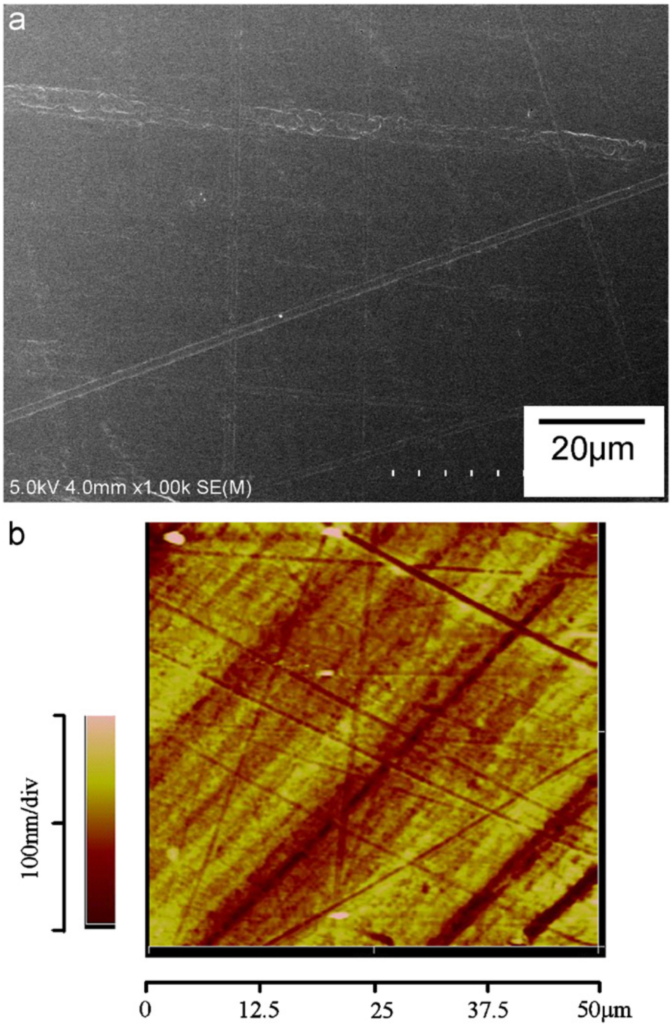

Fig. 5. Images of a ≈220 µg/cm^2 Ni film deposited on a polished NaCl substrate. (a) SEM image of a ≈220 µg/cm^2 Ni film on the polished NaCl substrate. (b) AFM image of a ≈220 µg/cm^2 Ni film floated from its polished NaCl substrate.

As collodion has proper elasticity and low crystallizability at ambient temperature, it is easy to get a smooth surface on the silicon wafer by the spin coater. It can be dissolved in an organic solvent such as acetone, but its solution rate is slow. When collodion serves as a parting agent, it makes the floating procedure last for a long time. At last, the Ni film would crinkle, sink or break due to the long time of surface tension action by the organic solvent. So the self-supporting collodion foil substrate (Fig. 6a) was adopted to shorten the solution period. The Ni foil made by this procedure is shown in Fig. 6b. It is smoother than the above shown four kinds of Ni foils as indicated in Fig. 6c. Its R_a decreases to 3.8 nm.

Designing smooth and removable substrates with proper elastic/plastic ratios was the key in obtaining smooth self-supporting Ni foils. The roughness of the substrates due to the crystallization of the parting agent limited the surface quality of the resulting Ni foils. Restriction against crystallization such as doping and control of the temperature was required for further research. At the same time,

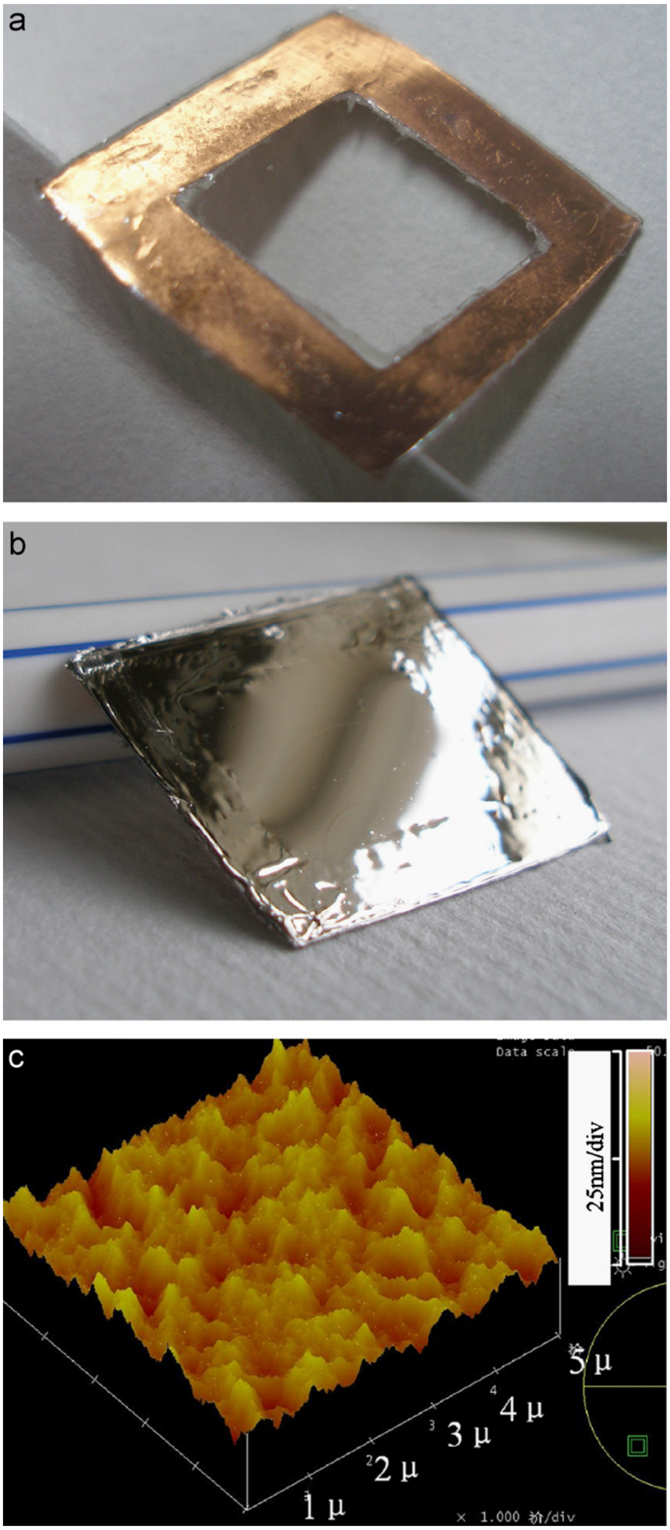

Fig. 6. Photos and AFM images of a self-supporting collodion film and a Ni film deposited on the collodion. (a) Photo of a self-supporting collodion film. (b) Photo of a Ni film on collodion. (c) AFM image of a $\approx 220\,\mu g/cm^2$ Ni film deposited on a self-supporting collodion film.

the desired substrates must have proper elastic/plastic ratios, so that wrinkles resulted from the relief of the stresses in the Ni film could be avoided. The intermolecular bonding (van der Waals) in collodion is weak, which permits individual molecules to readily slide past one another. Therefore, collodion easily can perform a smooth surface upon spin coating. And the long chain length makes collodion difficult to crystallize. Thus, the self-supporting collodion foils were appropriate substrates for the self-supporting Ni foils with low R_a.

4. Conclusions

The surface feature and mean roughness of the self-supporting Ni foils have close relationships with their substrates. Crystallized parting agents such as the betaine + sucrose and the potassium oleate make the Ni films deposited on them very rough. Soft parting agents (e.g., potassium oleate + sucrose) will result in the stress relief patterns on the Ni film. The polish treatment of NaCl substrate should be improved to reduce the effect of grinding marks on the film. A self-supporting organic film (e.g., collodion film) is the most suitable substrate for smooth Ni deposition. The R_a of such Ni films deposited on it is about 3.8 nm.

Acknowledgments

This research was sponsored by the National Natural Science Foundation of China (10576003).

References

[1] P. Maier-Komor, G. Dollinger, R. Krücken, Nucl. Instr. and Meth. A 561 (2006) 4.
[2] P. Maier-Komor, G. Dollinger, H.J. Körner, Nucl. Instr. and Meth. A 438 (1999) 73.
[3] P. Maier-Komor, G. Dollinger, C.M. Frey, H.J. Körner, Nucl. Instr. and Meth. A 362 (1995) 208.
[4] J.O. Stoner Jr., C.S. Jolivet, Nucl. Instr. and Meth. A 561 (2006) 11.
[5] J.O. Stoner Jr., Nucl. Instr. and Meth. A 521 (2004) 43.
[6] J.O. Stoner Jr., Nucl. Instr. and Meth. A 480 (2002) 44.
[7] J.O. Stoner Jr., Nucl. Instr. and Meth. A 397 (1997) 68.
[8] A. Stolarz, P. Maier-Komor, Nucl. Instr. and Meth. A 480 (2002) 194.
[9] A. Stolarz, J. Van Gestel, Nucl. Instr. and Meth. A 561 (2006) 115.
[10] V.Kh. Liechtenstein, T.M. Ivkova, E.D. Olshanski, R. Repnow, P. Steier, W. Kutschera, et al., Nucl. Instr. and Meth. A 561 (2006) 120.
[11] V.Kh. Liechtenstein, T.M. Ivkova, E.D. Olshanski, R. Golser, W. Kutschera, P. Steier, et al., Nucl. Instr. and Meth. A 521 (2004) 197.
[12] V.Kh. Liechtenstein, T.M. Ivkova, E.D. Olshanski, I. Feigenbaum, R. DiNardo, M. Döbeli, et al., Nucl. Instr. and Meth. A 397 (1997) 140.
[13] G.J. Xu, Y.Q. Wei, Nucl. Instr. and Meth. A 397 (1997) 62.
[14] P. Maier-Komor, Nucl. Instr. and Meth. 102 (1972) 485.
[15] T.M. Ivkova, V.Kh. Liechtenstein, E.D. Olshanski, Nucl. Instr. and Meth. A 362 (1995) 77.
[16] J.B. Héroux, X. Yang, W.I. Wang, J. Appl. Phys. 92 (2002) 4361.
[17] N. Bowden, W.T.S. Huck, K.E. Paul, G.M. Whitesides, Appl. Phys. Lett. 75 (1999) 2557.
[18] S.K. Basu, A.M. Bergstreser, L.F. Francis, L.E. Scriven, A.V. McCormick, J. Appl. Phys. 98 (2005) 63507.
[19] F. Iacopi, S.H. Brongersma, K. Maex, Appl. Phys. Lett. 82 (2003) 1380.