Characterization of electrodeposited Ni–Fe–SiC alloys for microelectromechanical applications

Xiaohu Zheng
Jiangsu Key Construction Laboratory of Numeric Machining Technology, Huaiyin Institute of Technology, Huaian 223003, China and MEMS and Nanotechnology Laboratory, School of Mechanical Systems Engineering, Chonnam National University, Gwangju 500-757, Republic of Korea

Xing Chen
MEMS and Nanotechnology Laboratory, School of Mechanical Systems Engineering, Chonnam National University, Gwangju 500-757, Republic of Korea

Feng Gu and YuanWei Liu
Jiangsu Key Construction Laboratory of Numeric Machining Technology, Huaiyin Institute of Technology, Huaian 223003, China

Dong-Weon Lee[a]
MEMS and Nanotechnology Laboratory, School of Mechanical Systems Engineering, Chonnam National University, Gwangju 500-757, Republic of Korea

Ni–Fe–SiC alloy is a promising material for the fabrication of microactuators. In this article, the electrolytic codeposition technique is used to deposit the Ni–Fe–SiC composite onto stainless-steel substrates, where nickel becomes alloyed with iron as the binder phase, and SiC becomes alloyed as dispersed particles. Analysis of the morphology indicates that the deposited SiC nanoparticles are compact, with the orientation of the deposited crystal planes indexed as (111), (200), (220), (311), and (222). The resistivity of the deposited SiC nanoparticles is about 30×10^{-8} Ω m. When the loading of Fe (wt %) ranges from 10% to 50% in the deposit, the electrodeposit shows a strong paramagnetism with a lowest value of coercivity of 2.75×10^{-2} A/m. In addition, the remanence shows a monotonic decrease with an increasing iron content in the deposit. It is demonstrated that the electroformed Ni–Fe–SiC alloy has better electromagnetic properties and a higher corrosion resistance (with a corrosion rate of 0.17 mg/dm^2 h 2*M* HCl) than the electroformed Ni–Fe alloy (with a corrosion rate of 0.23 mg/dm^2 h). © *2010 American Vacuum Society.*
[DOI: 10.1116/1.3498740]

I. INTRODUCTION

Ni–Fe deposits have a compact structure, a bright and smooth surface, and are widely used in the production of new materials that require specific mechanical, chemical, and physical properties.[1–3] These alloys also exhibit excellent properties of high hardness, thermal stability, and excellent magnetic properties. Various compositions of Ni–Fe alloys, such as permalloy (Ni–20% Fe) and invar alloy (Ni–64% Fe), can be used to produce microsensors, microactuators, and other microdevices.[4] Flynn[5] compared various microscale magnetic components and described a novel method for the manufacture and assembly of a Ni–20% Fe microinductor that is based on flip-chip bonding. The microcomponent fabricated with electroforming Ni–Fe alloy could be readily integrated with the other materials.[6,7] However, the corrosion resistance of the Ni–Fe deposit is lower than that of the electroformed Ni. Fortunately, SiC nanoparticles have been studied as a promising material for corrosion protection. Sharma[8] concluded that the Ni–Co–SiC composite coating shows a substantial improvement in hardness, as compared to pure Ni plating. Electroformed Ni alloys with SiC nanoparticles have a smaller grain size and finer properties than those of pure electroformed Ni. Moreover, SiC nanoparticles can improve the corrosion resistance of the deposit significantly.[9] Chou[10] revealed that the addition of SiC to the Ni–P alloy matrix can substantially reduce the residual stress in the deposit and therefore eliminate surface cracking. However, few researchers have studied the properties of electroformed Ni–Fe alloys with SiC nanoparticles, although this alloy promises to be widely used in the area of microfabrication.

In the present article, a Ni–Fe deposit, combined with nanocomposite SiC, has been synthesized and its electromagnetic properties were investigated. This article will be of potential benefit to a variety of the magnetic microactuators that require Ni–Fe–SiC electrodeposits.

II. EXPERIMENT

A. Experimental conditions

Ni–Fe deposits can be prepared from several different solutions. In this study, an acid sulfate solution containing

---

[a]Author to whom correspondence should be addressed; electronic mail: mems@chonnam.ac.kr

TABLE I. Composition and operating parameters for Ni–Fe–SiC composite coating.

Parameters	Value
$NiSO_4 \cdot 6H_2O$	250 g/l
$NiCl_2 \cdot 6H_2O$	30 g/l
H_3BO_3	40 g/l
$FeSO_4 \cdot 7H_2O$	3–7 g/l
$NaSO_4 \cdot 10H_2O$	10 g/l
SiC particles	2–3 g/l
Temperature of bath	55 °C
pH	<2.5
Current density	2.5–4 A/dm^2
Plating time	100–300 min
Stirring rate	600 rpm

(a)

(b)

FIG. 2. (Color online) (a) XRD profile of the deposited Ni–Fe–SiC alloy and (b) XRD profile of the pure Ni.

metal sulphates and some brightener was chosen. The main ingredients of the solution and the devices used in the experiment are as follows.

The main salts used were nickel and ferrous sulfate. The buffering and the stabilization of the solution were achieved by boric and citric acids, respectively. Sodium chloride, sodium sulfate, and some additives were used as anode activators and conductive agents. The SiC (2–3 g/l) had a grain size of 50 nm.

When preparing metal-salt solutions with different concentrations, the amount of nickel sulfate was fixed at 250 g/l, while the amount of iron was adjusted by the addition of various quantities of ferrous sulfate. Then the main electroforming parameters were set to be as follows: a pH value of the solution below 2.5, a current density from 2.5 to 4 A/dm^2, and a stirring rate of 500–700 rpm/min. The temperature of the solution was varied from 50 to 60 °C, and the concentration of $FeSO_4 \cdot 7H_2O$ from 3 to 7 g/l. In addition, several different process parameters were tested in the experiment. The composition and range of the experimental operating parameters are given in Table I.

B. Experimental setup

The experimental setup was as follows. The cathode was a stainless-steel (1Cr18Ni9Ti) plate. Before electroforming, the cathode was polished, and then its surface was activated in a 20% s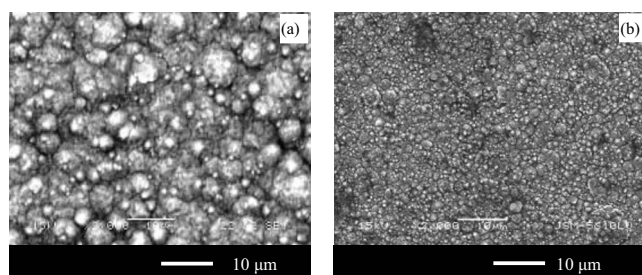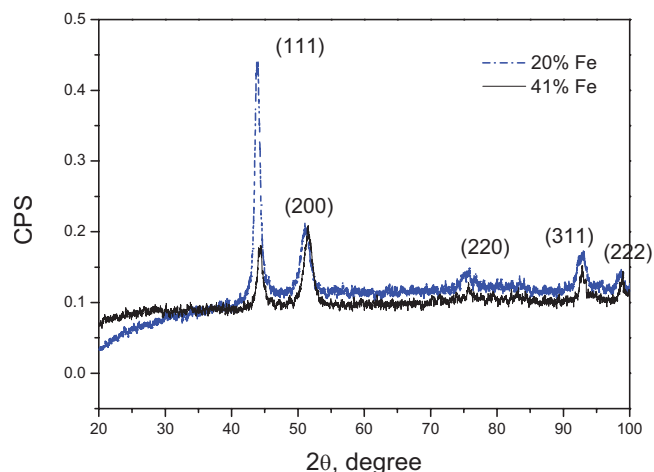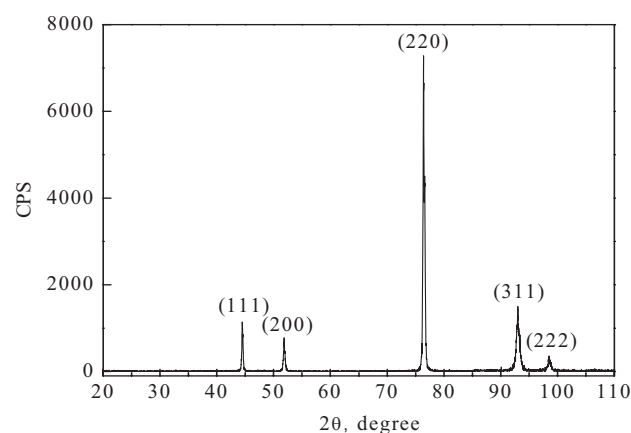ulfuric-acid solution. An electrical pure iron and an electrolyzed-nickel plate with an area ratio of Ni/Fe =10/1 were used as the anode, protected by a terylene pocket.

Scanning electron microscopy (SEM) images were obtained using a JSM-6300-type scanning electron microscope/energy spectrometer. A D/max-rc-type x-ray diffractometer was used to obtain x-ray diffraction (XRD) patterns. A Lakeshore 7307-9309-type vibrating-sample magnetometer was used to measure the magnetic properties of the sample. The iron component in the deposited alloys was measured by an energy-dispersive x-ray spectrometer.

III. RESULTS AND DISCUSSION

A. Surface-morphology investigation

It was found that the surface morphology of the deposit was sensitive to impurities in the solution, so some organic additives were used in the experiment to obtain a bright and smooth plating surface. Compared with pure Ni, a Ni–Fe–SiC deposit with a more compact structure was achieved in our experiment. Figure 1 reveals the surface morphology

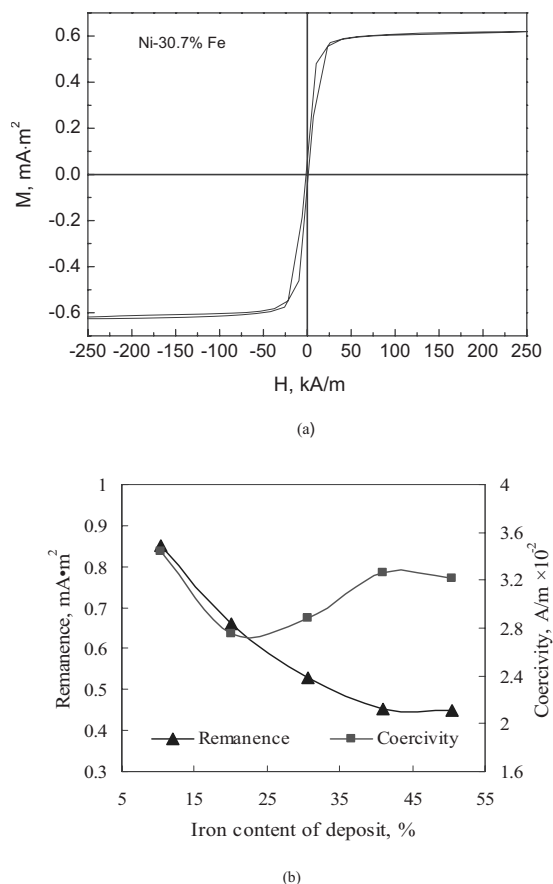FIG. 1. SEM images of deposited (a) Ni and (b) Ni–22.27% Fe–SiC.

FIG. 3. Magnetic properties of the deposits of (a) the hysteresis loop of the electroformed alloy and (b) the coercivity and remanence as a function of iron content in the deposits.

from a SEM photograph of the electrodeposited Ni and Ni–22.27% Fe–SiC. Subsequently, a corrosion test of the deposit was performed. The corrosion rate of the Ni–22.27% Fe–SiC deposit was 0.17 mg/dm^2 h in a $2M$ HCL solution, which was smaller than that of the electroformed Ni–20% Fe (0.23 mg/dm^2 h). The results indicate that adding the SiC particles significantly improved the corrosion rate of the deposit. During the deposition process, the rate was kept at about $5-8$ μm/h. After being removed from the stainless-steel substrate by mechanical cleavage and exfoliation, the electrodeposits showed no curling or buckling. That indicates low internal stress in the deposits.

As shown in Fig. 2(a), the plating has a well-organized crystal structure. X-ray diffraction peaks were observed at (111), (200), (220), (311), and (222). When the iron content changed from 20% to 41%, the most intense diffraction peak changed from (111) to (200). As shown in Fig. 2(b), pure Ni has diffraction peaks at (111), (200), (220), (311), and (222). The most intense diffraction peak is (220).[11] The change of the crystalline orientation may be attributed to the SiC particles and the change of the iron concentration. Some inhibi-

tors, such as H_2 and $Ni(OH)_2$ in the interface between the cathode and solution, not only affected the plating morphology and the selective adsorption but also altered the crystal growth mode.

B. Electromagnetic properties

To determine the magnetic properties of the deposit, the hysteresis loop of the Ni–30.7% Fe–SiC deposit was recorded. As shown in Fig. 3(a), the Ni–30.7% Fe–SiC deposit has superparamagnetic characteristics with a remanent magnetization of 0.528 mA m^2 and a coercivity of 2.88 $\times 10^{-2}$ A/m. Ni–20% Fe–SiC has the smallest coercivity value of 2.75×10^{-2} A/m, which was less than that of the IJ85 permalloy.[12] The saturated magnetization of the deposit was only 10% of the IJ85 permalloy. Comparisons of the remanence and coercivity of the deposits are shown in Fig. 3(b). The Ni–20% Fe–SiC deposit has the lowest coercivity, and the remanence shows a monotonic decrease with an increasing iron content in the deposit. This demonstrates that the deposit shows strong paramagnetism and has better soft magnetic properties than that of the IJ85 permalloy. The electrical resistivity of the deposit was about 30 $\times 10^{-8}$ Ω m for iron contents ranging from 10% to 50%.

IV. CONCLUSION

Ni–Fe–SiC electrodepositing technology and the physical properties of the resulting deposits are discussed in this study. It is shown that the Ni–Fe–SiC electrodeposit has excellent electromagnetic properties, compact structure, and the SiC particles significantly improved the corrosion resistance of the Ni–Fe deposit. Therefore, it is a promising material for microfabrication.

ACKNOWLEDGMENTS

[1]F. J. Czerwinski, J. Electrochem. Soc. **143**, 3327 (1996).
[2]K. Msellak and J. P. Chopart, J. Magn. Magn. Mater. **281**, 295 (2004).
[3]G. Zhan-Cheng, Chin. J. Nonferrous Met. **14**, 273 (2004).
[4]Y. Zhuang, M. Vroubel, and B. Rejaei, Solid-State Electron. **51**, 405 (2007).
[5]D. Flynn, J. Phys.: Conf. Ser. **34**, 112 (2006).
[6]A. D. Yalcinkaya, U. Hakan, and H. Sven, IEEE Photonics Technol. Lett. **19**, 330 (2007).
[7]S. D. Leith and D. T. Schwartz, J. Microelectromech. Syst. **8**, 384 (1999).
[8]G. Sharma, R. K. Yadava, and V. K. Sharma, Bull. Mater. Sci. **29**, 491 (2006).
[9]C. T. J. Low, R. G. A. Wills, and F. C. Walsh, Surf. Coat. Technol. **201**, 371 (2006).
[10]M.-C. Chou, M.-D. Ger, and S.-T. Ke, Mater. Chem. Phys. **92**, 146 (2005).
[11]K. P. Wong, K. C. Chan, and T. M. Yue, J. Mater. Process. Technol. **117**, 97 (2001).
[12]X.-H. Zheng, D. Zhu, and N.-S. Qu, Mater. Mech. Eng. **31**, 33 (2007).