Electrochemical and Solid-State Letters, **2** (12) 624-626 (1999)
S1099-0062(99)07-005-4 CCC: $7.00 © The Electrochemical Society, Inc.

A NiFeMo Electroplating Bath for Micromachined Structures

William P. Taylor,[a,*,c] Michael Schneider,[b,*] Henry Baltes,[b,*] and Mark G. Allen[a,z]

[a]*Georgia Institute of Technology, School of Electrical and Computer Engineering, Atlanta, Georgia 30332-0250, USA*
[b]*Physical Electronics Laboratory, ETH Zurich, 8093 Zurich, Switzerland*

A number of micromachined magnetic sensors and actuators require materials with desirable magnetic properties such as high permeability and thicknesses in the range of several micrometers. Materials with appropriate magnetic properties have been reported for magnetic storage applications, but often in thickness ranges insufficient for micromachined sensors and actuators. We report the results of an electroplating bath for the deposition of a high permeability, low coercivity material, a NiFeMo alloy. Using this bath, the NiFeMo alloy may be electrodeposited up to 5 μm in thickness. The residual stress of the electroplated NiFeMo material remains larger than that for NiFe (Permalloy) electrodeposits.

Manuscript submitted July 1, 1999; revised manuscript received September 13, 1999. Available electronically October 15, 1999.

The development of micromachined magnetic devices has relied primarily on the use of nickel-iron Permalloy. Permalloy is used in a number of applications since it has good soft magnetic properties, high permeability, high magnetoresistive effect, low magnetostriction, stable high frequency operation, and excellent mechanical properties.[1] In hard disk magnetic recording heads, Permalloy is widely used for magnetoresistive sensors and flux guiding elements. Devices such as magnetic separators,[2] micropumps,[3] magnetic micromotors,[4-6] inductors,[7] switches,[8] and microrelays[9] have also been fabricated using Permalloy as the magnetic material as well as moving members. Permalloy microstructures have been used as flux guides for sensitivity improvement of magnetotransistors[10] and as a ferromagnetic core in mircofluxgate sensors.[11] These structures can be integrated with complementary metal oxide semiconductor (CMOS) circuitry on a single chip.

However, there remains a need to improve the magnetic properties of the materials used in such applications. If the permeability of the films used in magnetic microactuators can be increased, the efficiency of the actuator may also increase. In addition, large permeability may increase the sensitivity of many magnetic sensors. Large saturation flux density allows devices to be made smaller, or it may allow for increased actuator force, and allow devices such as transformers to store magnetic energy more efficiently. In many applications for magnetic sensors, the coercivity of the material should be as low as possible. Thus, the development of high permeability, and large saturation flux density materials may allow for further improvements in or new types of magnetic actuators.

The large body of literature on magnetic materials developed for magnetic storage applications provides many insights for magnetic microelectromechanical systems (MEMS). However, MEMS applications generally require the magnetic material to have greater thickness when compared to magnetic storage applications. The thickness requirement may result from several design constraints. The required structural integrity of the MEMS actuator may place a minimum thickness on the order of tens of micrometers. For the magnetic MEMS actuator, the saturation flux density, $\mathbf{B}_s$, must also be considered. In order to achieve the ability to carry a magnetic flux, Φ, the cross-sectional area, A, of the actuator must be sized according to

$$\Phi = \mathbf{B}_s A \qquad [1]$$

The cross-sectional area, A, is the linewidth of the pattern of interest times the thickness of the magnetic material. As it may be desirable to reduce the linewidth of the device, in order to increase the number of devices on a substrate or to reduce the overall size of the device, the designer could increase the thickness, t.

Previously, Permalloy (NiFe 80/20) has been the most widely used electroplated material in magnetic microdevices (see, for example, Ref. 2-12). The reported relative permeability (without correcting for demagnetization effects) of these films is generally on the order of 500-1000, with coercivity ranging from 1-5 Oe. Some research into alternative materials has begun, *e.g.*, a NiCo alloy of 79/21 has been reported with relative permeability of 19.4.[13]

Table I. Electroplating bath composition.

Chemical	NiFe bath[16] g/L	NiFeMo bath g/L
$NiSO_4 \cdot 7H_2O$	200	60
$NiCl_2 \cdot 6H_2O$	5	—
$FeSO_4 \cdot 6H_2O$	8	4
$Na_2MoO_4 \cdot 2H_2O$	—	2
H_3BO_3	25	—
NaCl	—	10
Citric acid	—	66
Saccharin	3.0	3.0

Another alloy that has been reported in the literature is NiFeMo.[14,15] The original bath was reported in 1965,[14] and only investigated films up to hundreds of angstroms in thickness. In this case, the materials were not electroplated to the thickness required for MEMS applications, due, in some cases, to large internal stresses in the deposited films. In spite of this, the previous data can be used as a starting point for the development of new electroplating baths.

Recent work has reported electroplated films up to 1 μm thick,[15] however, these films are still not thick enough for some micromachining applications. These films in a single layer consisted of Ni-17Fe-4Mo, and had coercivities on the order of 0.06 Oe, with saturation magnetization of 0.72 T and a relative permeability of 3400 at 10 MHz. The baths developed in this work are capable of plating films up to and in excess of 5 μm thick.

The purpose of this work was to develop a method for the electrodeposition of NiFeMo films of thicknesses sufficient to be of interest in micromachining applications, and to assess the magnetic properties and mechanical properties of these very thick electrodeposited films.

Experimental

The electroplating baths were developed based on the previous baths in the literature. A standard nickel-iron plating bath was used for relative residual stress comparison.[16] The bath compositions for the NiFe and NiFeMo films deposited in this study are given in Table I. A major difference between the NiFeMo electroplating bath reported in Ref. 14 and the NiFeMo electroplating bath reported here was an increase in the saccharin concentration. No pH adjustment was subsequently made after the additional saccharin was used. The motivation behind the addition of saccharin was to reduce the

---

[*] Electrochemical Society Active Member.
[c] Present address: Teledyne Electronic Technologies, Los Angeles, CA 90066, USA
[z] E-mail: mallen@ee.gatech.edu

amount of residual stress in the films, and thereby allow thicker films to be plated. This effect has been reported by other authors[17] for Permalloy electroplating baths.

Samples were plated on glass substrates with a titanium and gold seed layer. The NiFeMo alloy was also plated on a polished silicon substrate with a titanium and copper seed layer. Two current densities were investigated: 10 and 30 mA/cm^2, with a typical nickel anode size of 50 x 50 mm. No heating was employed during the electroplating processes described, and only gentle stirring was used.

A Lakeshore LS7300 vibrating sample magnetometer was used to obtain magnetization *vs.* applied field curves for the films. The **B-H** curves (described below) were then resolved in accordance with standard magnetic analysis techniques.[18] The applied field, **H**, is measured directly by a built-in field sensor. The magnetic induction, **B**, may be obtained (for cgs units) by dividing the resultant moment measurements by the sample volume, multiplying by 4π, and adding the applied field. The effective permeability of the material may then be calculated for cgs units as the ratio of **B** to **H** for a given point on the **B** *vs.* **H** curve.

The most critical procedure in determining relative permeability and saturation flux density is the measurement of the volume of the thin films. Several approaches to this may be taken. The sample may be measured on a mass balance and the volume determined for a material of known density. However, since the density of the plated alloy is not precisely known, this method may not be accurate. The method employed here was to measure the thickness for a sample of known area. Since the sample patterns are made using photolithography techniques, the amount of error in the area to be plated is generally small, and does not vary significantly from sample to sample. Determining the actual height of the sample introduces the largest component of error in the volume measurement. Films may have thickness nonuniformities, as well as nodules and grains, both of which reduce the accuracy of the volume measurement. By using a surface profilometer, the height can easily be determined to within 0.5 μm. However, if the films are only 3 μm thick, this results in a 15-20% error. On some samples, an optical profilometer Microfocus UBM model was used to measure the thickness. Two-dimensional scans were taken over the complete film area and the film volume was calculated by integration. This technique still has a ±10% error, but a much more accurate description of the surfaces is obtained. Nodules and grains are included more accurately in this method, and thus, the error in the volume measurement is reduced. Note that the values of saturation flux density and relative permeability reported here contain these volume measurement uncertainties.

The residual stress of the plated NiFeMo films was measured using a Flexus machine. Films were plated on a single-crystal silicon substrate, the resultant curvature of which can be used to assess film stress. For example, by measuring the radius of curvature of a silicon wafer bearing a seed layer, and then measuring the radius of curvature of the wafer after electroplating, the stress of the deposited layer can be determined. The resultant residual stress calculation depends on the measured thickness of the electroplated film. Since the film thickness may vary across the surface of the wafer, these results should be used as a qualitative indication of stress in the films. The stress of the NiFeMo films was approximately 2-3 times greater than that observed in NiFe films of the same thickness. All comparisons for stress were made for films deposited at room temperature without the addition of an applied magnetic field during plating. The effect of magnetic fields applied during plating on the mechanical stress of the film was not investigated in this work.

Magnetic Properties

During initial plating experiments for the NiFeMo system, it was determined that by plating in a magnetic field, the coercivity of the film could be reduced, and the field required for saturation could also be reduced. A similar improvement in magnetic properties has been reported previously[19,20] for other materials. Since the properties of this alloy which are of most interest are low coercivity and high permeability, only the magnetic properties of the NiFeMo films plated in a magnetic field are discussed.

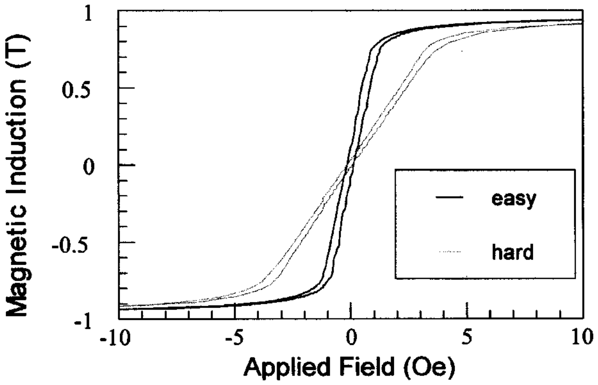

Figure 1. Nickel-iron-molybdenum magnetic induction *vs.* applied magnetic field for easy and hard axis determined by vibrating magnetometer.

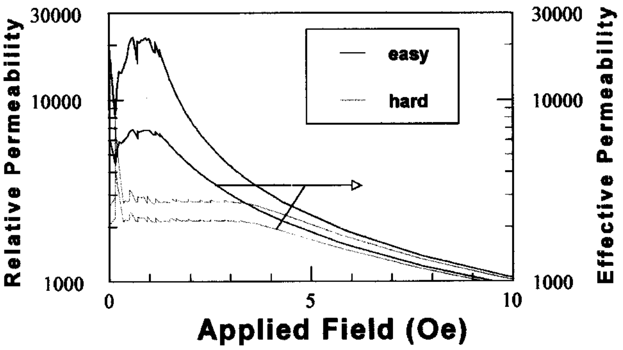

Figure 2. Nickel-iron-molybdenum relative permeability as derived from effective permeability measurement. The relative permeability is shown after correction for the film demagnetization effect[21] by ellipsoidal approximation. The effective permeability is the as-measured, uncorrected relative permeability.

Two different current densities were used for plating experiments, 10 and 30 mA/cm^2. Analysis of the atomic composition of the films at 30 mA/cm^2 was performed using energy dispersive spectroscopy (EDS); the atomic content of the films plated without the application of a magnetic field was 85Ni-14Fe-1Mo. With the application of a magnetic field, the films showed a tendency towards increased nickel and molybdenum concentrations, and a decrease of iron content.

Square samples of NiFeMo films were plated at 10 mA/cm^2 under the influence of a 50 mT magnetic field applied with a Helmholtz coil. One side of the square sample was aligned parallel to the axis of the applied magnetic field. This axis was then referred to as the in-plane magnetic easy axis. The second in-plane axis of the square and the thickness of the magnetic material sample were both positioned perpendicular to the applied field during plating. The in-plane axis of the square perpendicular to the applied field is then the in-plane magnetic hard axis. An example of the typical magnetic induction *vs.* applied field curve is shown in Fig. 1. The measured $\mathbf{B}_{\mathrm{sat}}$ values ranged from 0.97 to 1.07 T in both the easy and hard axis. Measured values of effective permeability were as large as 7000 for the easy axis (the in-plane direction of the film parallel to the applied magnetic field during plating) and 2200 for the hard axis (the in-plane direction of the film perpendicular to the magnetic field during plating).

The measured data were corrected for demagnetization according to the actual geometric film parameters using an ellipsoidal approximation.[21] Figure 2 shows the resulting relative permeability *vs.* applied field for the NiFeMo films. After correcting for demagnetization, maximum relative permeabilities, on the order of 20,000 for the easy axis and almost 3000 for the hard axis, are observed. These films showed the lowest coercivity of any of the films, which were in the range of 0.35 and 0.10 Oe for the easy and hard axis, respectively.

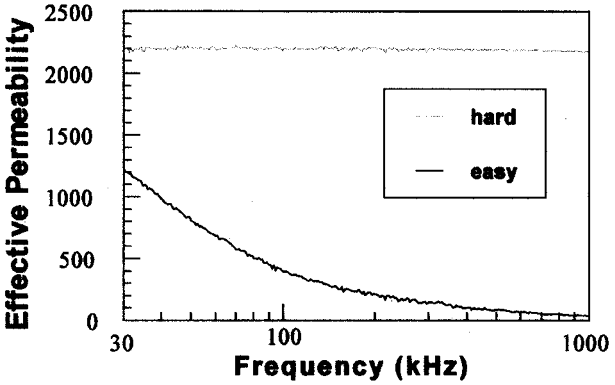

Figure 3. Nickel-iron-molybdenum effective permeability *vs.* frequency of applied magnetic field. The hard axis permeability is constant up to 1 MHz.

The ac magnetic properties of the NiFeMo films were also investigated. This was achieved by measuring the impedance change of a U-shaped ferrite core while closing the core gap with a sample of the NiFeMo film. Figure 3 shows the easy and hard axis effective permeability *vs.* frequency. The hard axis effective permeability is constant over the entire frequency range from 30 kHz up to 1 MHz and matches well with the VSM-determined low frequency value of 2200. Such results demonstrate the usefulness of this material for high frequency sensors. As expected, the easy axis permeability strongly decreases with frequency. This particular NiFeMo electroplating bath has been used in the development of new micromachined magnetic sensors[22,23] and high frequency inductive components.[24]

Conclusions

A NiFeMo magnetic material has been successfully electrodeposited in thickness ranges useful for many micromachining applications. An electroplating bath capable of plating NiFeMo films up to 5 μm in thickness has been developed. Films plated from this bath show reduced coercivity and higher effective permeability when compared to other films commonly used for micromachining magnetic applications. Such films have the potential to increase the efficiency of microactuators, and increase the sensitivity of magnetic microsensors such as microfluxgate sensors.

Acknowledgments

The authors thank Charles Chung, Dr. Paul Kohl, and Dr. Nicole Grove of Georgia Tech and Dr. Oliver Brand, currently at the ETH Zurich, for their technical discussion and assistance. The authors also thank other colleagues at both Georgia Tech and ETH Zurich for their help and technical discussion.

The Georgia Institute of Technology assisted in meeting the publication costs of this article.

References

1. J. Mallinson, *Magneto-Resistive Heads-Fundamentals and Applications,* Academic Press, New York (1996).
2. T. M. Liakopoulos, J. W. Choi, and C. H. Ahn, in *Proceedings of Transducers ‘97,* p. 485 (1997).
3. W. Zhang and C. H. Ahn, in *Proceedings of the Solid State Sensor and Actuator Workshop,* p. 94 (1996).
4. B. Wagner, M. Kreutzer, and W. Benecke, in *Proceedings of the IEEE Microelectromechanical Systems Workshop,* p. 183, IEEE, Piscataway, New Jersey (1992).
5. C. H. Ahn, Y. J. Kim, and M. G. Allen, in *Proceedings of the IEEE Microelectromechanical Systems Workshop,* p. 1, IEEE, Piscataway, NJ (1993).
6. H. Guckel, K. J. Skrobis, T. R. Christenson, J. Klein, S. Han, B. Choi, E. G. Novell, and T. W. Chapman, in *Proceedings of Transducers ‘91* (1991).
7. C. H. Ahn, Y. J. Kim, and M. G. Allen, in *Proceedings of Tansducers ‘93,* p. 70 (1993).
8. J. A. Wright and Y. C. Tai, in *Proceedings of the Twelfth IEEE International Conference on Micro Electro Mechanical Systems,* p. 594, IEEE, Piscataway, NJ (1999).
9. W. P. Taylor, M. G. Allen, and C. R. Dauwalter, in *Proceedings of the Solid State Sensor and Actuator Workshop,* p. 231 (1996).
10. M. Schneider, R. Castagnetti, M. G. Allen, and H. Baltes, in *Proceedings of the IEEE Microelectromechanical Systems Workshop,* p. 151, IEEE, Piscataway, NJ (1995).
11. S. Choi, S. Kawahito, Y. Matsumo, M. Ishida, and Y. Tadokoro, *Sens. Actuators A,* **55**, 121 (1996).
12. T. R. Ohnstein, J. D. Zook, H. B. French, H. Guckel, T. Earles, J. Klein, and P. Mangat, in *Proceedings of the Solid State Sensor and Actuator Workshop,* p. 196 (1996).
13. B. Loechel and A. Maciossek, *J. Electrochem. Soc.,* **143**, 3343 (1996).
14. W. O. Freitag and J. S. Mathias, *J. Electochem. Soc.,* **112**, 64 (1965).
15. O. Shinoura, *Denki Kagaku oyobi Kogyo Butsuri Kagaku,* **63**, 473 (1995).
16. A. B. Frazier and M. G. Allen, *J. Microelectromech. Syst.,* **2**, 87 (1993).
17. K. Lin, J. W. Chang, and L. T. Romankiw, in *Magnetic Materials, Processes, and Devices IV,* L. T. Romankiw and D. E. Herman, Jr., Editors, p. 626, The Electrochemical Society Proceedings Series, Pennington, NJ (1995).
18. Users manual, Lakeshore Cryotronics Inc., model 7300 VSM.
19. Anderson, Castellani, McCaffrey, and Romankiw, U.S. Pat. 4,003,768.
20. T. Osaka, T. Homma, K. Saito, A. Takekoshi, Y. Yamazaki, and T. Namikawa, in *Magnetic Materials, Processes, and Devices,* L. T. Romankiw and D. E. Herman, Jr., Editors, p. 267, The Electrochemical Society Proceedings Series, Pennington, NJ (1992).
21. D. Craik, *Structure and Properties of Magnetic Materials,* Pion Limited, London (1979).
22. M. Schneider, S. Kawahito, Y. Tadokoro, H. Baltes, *1997 Tech. Dig. Int. Electron Devices Meet.,* p. 907 (1997).
23. R. Steiner, M. Schneider, F. Mayer, U. Munch, M. Mayer, and H. Baltes, in *Proceedings of the Microelectromechanical Systems Workshop,* p. 603, IEEE, Piscataway, NJ (1998).
24. J. Park, W. Taylor, M. Allen, S. Han, in *APEC ‘98-Thirteenth Annual Applied Power Electronics Conference and Exposition,* Anaheim, p. 379 (1998).