A NiFeMo Electroplating Bath for Micromachined Structures

William P. Taylor,a,* Michael Schneider,b,* Henry Baltes,b,* and Mark G. Allena,z

aGeorgia Institute of Technology, School of Electrical and Computer Engineering, Atlanta, Georgia 30332-0250, USA
bPhysical 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.

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Table I. Electroplating bath composition.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>NiFe bath 16 g/L</th>
<th>NiFeMo bath g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiSO₄·7H₂O</td>
<td>200</td>
<td>60</td>
</tr>
<tr>
<td>NiCl₂·6H₂O</td>
<td>5</td>
<td>—</td>
</tr>
<tr>
<td>FeSO₄·6H₂O</td>
<td>8</td>
<td>4</td>
</tr>
<tr>
<td>Na₂MoO₄·2H₂O</td>
<td>—</td>
<td>2</td>
</tr>
<tr>
<td>H₂BO₃</td>
<td>25</td>
<td>—</td>
</tr>
<tr>
<td>NaCl</td>
<td>—</td>
<td>10</td>
</tr>
<tr>
<td>Citric acid</td>
<td>—</td>
<td>66</td>
</tr>
<tr>
<td>Saccharin</td>
<td>3.0</td>
<td>3.0</td>
</tr>
</tbody>
</table>

The large body of literature on magnetic materials developed 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, however, these films are still not thick enough for some micromachining applications. These films in a single layer consisted of NiFeMo electroplating bath reported here. The purpose of this work was to develop a method for the electrophotogravimetry 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. 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

[1] The cross-sectional area, A, of the actuator must be sized according to

\[ \Phi = B_s A \]
amount of residual stress in the films, and thereby allow thicker films to be plated. This effect has been reported by other authors 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/cm², 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. 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 \( 4\pi \), 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 \( \mu \)m. However, if the films are only 3 \( \mu \)m 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 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 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.

Two different current densities were used for plating experiments, 10 and 30 mA/cm². Analysis of the atomic composition of the films at 30 mA/cm² 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/cm² 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 \( B_{sat} \) 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. 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.
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\textsuperscript{22,23} and high frequency inductive components\textsuperscript{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 \textmu m 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.

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