Study on the Electroforming Ni-Co-Al2O3 Alloy

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ABSTRACT. Electrolytic codeposition technique was adopted in the deposition of Ni-Co-Al2O3 coating on the stainless substrate, using nickel alloyed with iron as the binder phase with Al2O3 as dispersed particles. The results indicated that the deposit with Al2O3 nanoparticles was level and compact; the crystal-planes of the deposit were (111), (220) and (200). The resistivity of deposit was about 30 μΩ·cm. when the Co(wt.%) ranged from 10% to 50% in the deposit, the electrodeposit Ni-20%Co-Al2O3 has a strong paramagnetism effect with the smallest coercivity of 2.75×10⁻² A/m. The remanence showed a monotonic decrease with the increasing iron content in deposit. Which proved that the electroformed NiCo-Al2O3 alloy has good electromagnetic property and higher corrosion resistance than those of electroforming Ni-Co alloy. It is a promising material in the fabrication of micro sensor.

Introduction

NiCo deposit has a compact structure, bright and smooth surface and was widely used in the production of new materials that required specific mechanical, chemical and/or physical properties[1,2,3]. These alloys also exhibit excellent properties of levelling, high hardness, thermally stable, and fine magnetic properties. Various compositions of Ni-Co alloys such as Permalloy (Ni-20%), Invar alloy (Ni-64%Fe) and high strength Ni–Fe alloy can be used to produce micro sensor, micro actuators and other MEMS device[4,5]. David Flynn compared various microscale magnetic components and described a novel method to manufacture and assemble a Ni-20%Fe microinductor that is based on flipchip bonding[6]. The micro component fabricated with electroforming Ni-Co alloy could be integrated with other materials easily[7,8]. But the corrosion resistance of Ni-Co deposit was lower than that of the electroforming Ni. Al2O3 nano particles has been studied as a promising material in corrosion protection. Gajendra Sharma concluded that the Ni–Co–Al2O3 composite coating shows substantial improvement in the hardness as compared to pure Ni plating[9]. Electroforming Ni alloys with nano Al2O3 has a smaller grain size and finer property than that of the electroforming Ni, moreover, nano Al2O3 could improve the corrosion resistance of the deposit considerably[10]. Min-Chieh Choua revealed that adding SiC to the Ni–P alloy matrix substantially reduces the residual stress in the deposit and, therefore, eliminates surface cracking[11].

In the present work, the nickel-iron deposit with nanocomposites Al2O3 and its electromagnetic property were investigated. The work will be benefit to the fabrication of many magnetic microactuators with Ni-Co-Al2O3 electrodeposits.
Experimental procedure

There are many kinds of solution for Ni-Co deposit. An acid sulphate solution, containing metal sulphates and some brightener, was chosen for this study [4]. The main content of solution and devices using in the experiment was presented as follows:

Main Salts: Nickel Sulphate, Ferrous Sulfate. Buffer Solution and Stabilizer: Boric acid, Citric acid. Anode activators and conductive agent: Sodium Chloride, Sodium Sulphate and some additive, 50nm grain size Al2O3 nanoparticles :2-3g/L. When preparing metal salt solutions of different concentrations, the amount of Nickel Sulphate was 250g/l, the amount of iron ion was adjusted by the Ferrous Sulfate additives. The main electroforming parameters: PH value of the solution was less than 2.5, current density was set from 2.5 to 4 A/dm$^2$, stirring velocity: 500-700 rpm/min, the solution temperature was ranged from 50 to 60 $^\circ$C, CoSO$_4$·7H$_2$O: 3-7g/l, several different process parameters were adopted in the experiment. The composition and range of experimental operating parameters were given in table 1.

<table>
<thead>
<tr>
<th>Parameter for equipment setup</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature of bath</td>
<td>55 $^\circ$C</td>
</tr>
<tr>
<td>PH</td>
<td>&lt; 2.5</td>
</tr>
<tr>
<td>Current density</td>
<td>2.5-4A/dm$^2$</td>
</tr>
<tr>
<td>Plating time</td>
<td>100-300 min</td>
</tr>
<tr>
<td>Stirring rate</td>
<td>600 rpm</td>
</tr>
</tbody>
</table>

Table 1  Operational parameters of Ni-Fe-Al2O3 composite

| NiSO$_4$·6H$_2$O | 250g/l |
| NiCl$_2$·6H$_2$O | 30g/l |
| H$_3$BO$_3$      | 40g/l |
| CoSO$_4$·7H$_2$O | 3-7g/l |
| NaSO$_4$·10H$_2$O| 10g/l |
| Al2O3 particles  | 2-3g/l |

Results and discussion

The properties of Ni-Co deposit are affected by several factors such as the texture, compositions [12], etc. The main properties of the plating were presented as follows:

Surface morphology of the plating

The deposit surface morphology was sensitive to the impurity in the solution. Some organic additives were adopted in the experiment to obtain the bright and smooth plating surface. Ni-Co-Al2O3 deposit has a more compact structure than that of pure Ni. Fig.1 was the surface morphology SEM photograph of the electrodeposit Ni and Ni-22.27%Co-Al2O3. The corrosion test of the deposit was performed subsequently. The corrosion rate of Ni-22.27% Co -Al2O3 deposit was 0.17 mg/dm$^2$ per hour in 2M HCL solution, which was smaller than that of the electroforming Ni-20% Co (0.23 mg/dm$^2$ per hour). The results indicated that the corrosion rate of the deposit be improved significantly with the ad Al2O3 particles. The deposition rate is about 5μm-8μm per hour. The electrodeposits showed no curling or buckling after removing from the substrate. Which indicated that internal stresses was relatively low.

Fig. 2 showed the X-ray diffraction of electroforming Ni-Co-Al2O3 alloy. The plating has a well
The X-ray diffraction peak were (111), (220) and (200). When the Co content reached to 41%, the highest diffraction peak fall from (111) to (220). Pure Ni has a diffraction peak of (111). The change of the crystalline orientation may be attributed to the cathodic over-voltage and the change of the ion concentration. There are some inhibition material such as H2, Ni(OH)2 on the cathode-solution interface. They not only affected the plating morphology and the selective adsorption, but also altered the crystal growth mode.

**Electromagnetic property**

To compare the magnetism property with permalloy, the hysteresis loop of Ni-30% Co-Al2O3 deposit and permalloy (IJ85) were tested under the same condition. As showed in Fig.3, Ni-30% Co-Al2O3 deposit has superparamagnetic characters with magnetic remnant was 0.528 mA·m², coercivity was $2.88 \times 10^{-2}$ A/m. Moreover Ni-20% Co-Al2O3 has a smallest coercivity value of $2.75 \times 10^{-2}$ A/m, which was less than that of IJ85 permalloy. Saturated Magnetization value of deposit was only 10% of the IJ85 alloy. The remanence and coercivity comparison of the deposit was showed in Fig. 4. Ni-20%Fe-Al2O3 deposit has a lowest remanence, and the coercivity show a monotonic decrease with increasing the iron content in deposit. The test data indicate that the deposit has a satisfied magnetism property. The resistivity of deposit was about $30 \mu \Omega \cdot \text{cm}$, when the iron content ranged from 10% to 50% in the deposit.
Conclusions

Ni-Co-Al2O3 electrodeposit technology and physical property was discussed in this paper. The hysteresis loop of electroforming alloy and the trend of the iron content in the deposit against the coercivity, remanence were given. From the experiment it can concluded that the Ni-Co-Al2O3 electrodeposit has fine electromagnetic property, high corrosion resistance, compact structure, and could be used in MEMS actuator manufacture.

References