

Electrodeposited Fe-Ni Films Prepared from a Tartaric-acid-based Bath

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Recently, we reported a citric-acid-based plating bath is one of the hopeful plating baths for obtaining Fe-Ni films with good soft magnetic properties. In this report, hydroxylic acid of tartaric acid, which belongs to the same acid group at citric acid, was applied to prepare the films, and the effects of tartaric acid on the magnetic and the structural properties were investigated. Although the overall trend of the effects obtained for tartaric acid resembles our previous results for the citric-acid-based bath, the cathode efficiency for the tartaric-acid-based bath shows a slightly higher value as compared with that for the citric-acid-based bath. From these results, we conclude that the tartaric-acid-based bath is also an environmentally friendly plating bath.

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I. INTRODUCTION

Fe-Ni alloys are known for their exceptionally good soft magnetic properties, such as the high permeability and low coercivity, and their films have been applied in electric devices for the purpose of shielding and converging magnetic flux. The Fe-Ni films in the devices are commonly prepared by using electrodeposition methods because the methods have many productive advantages, such as high economic viability of the process without the need for a vacuum. Many studies have been carried out on electrodeposited Fe-Ni films, and good soft magnetic properties of the films have been reported [1-3]. In previous reports on Fe-Ni plated films, boric acid was most commonly used to control the pH of the plating bath. Although the boric acid played an important role in the plating process, boron and its compound in the waste bath were specified as toxic substances by an Ordinance of the Ministry of the Environment in Japan. Hence, in order to remove boron from the plating bath, we have explored possible alternatives to boric acid. Recently, we have found that a citric-acid-based bath is a good plating bath for obtaining Fe-Ni films with good soft magnetic properties [4]. Here, we focus on tartaric acid, which is in the same acids group as citric acid [5]. In the present study, the magnetic and the structural properties of Fe-Ni films prepared from a tartaric-acid-based bath have been investigated.

II. Experimental Procedures

Preparation of Electrodeposited Fe-Ni films

The base electrolyte of the plating bath contained the following: 275 g/L of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 35 g/L of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 50 g/L of NaCl and 5 g/L of $\text{C}_7\text{H}_4\text{NNaO}_3\text{S} \cdot 2\text{H}_2\text{O}$. We added 0-300 g/L of L(+)-tartaric acid ($\text{C}_6\text{H}_6\text{O}_6$) to the above-mentioned plating bath. The pH in the plating bath was not adjusted. Five-hundred- μm -thick Ni and Cu plates were used as electrodes, and 75- mm^2 Fe-Ni films were electrodeposited on the cathode of the Cu plate. The temperature of the plating bath was kept at 50°C during the deposition. A current density of 2 mA/mm^2 and a deposition time of 10 min were employed,

and these conditions were controlled by using a computer-aided dc current source.

Measurements

The magnetic properties of the electrodeposited Fe-Ni films were evaluated from the dc hysteresis loops. In order to obtain the loops, we operated a B-H tracer (Riken Denshi BHS-40) at a field sweep rate of 50 mHz and a maximum excitation field of approximately 4 kA/m. The thicknesses of the Fe-Ni films were measured every 7 mm² (12 points) with a micrometer (Mitutoyo CPM15-25MJ) and determined by averaging the measured values. The crystal structures of the films were analyzed by using X-ray diffraction (XRD) with the CuK α radiation (RigakuRint 2000). The compositions of the films were evaluated on a Hitachi High-technologies S-3000 spel out SEM-EDX system. The compositions of the 75-mm² Fe-Ni films were determined by averaging the values obtained from approximately 9-mm² areas at 9 different points in each film. All measurements were carried out for the Fe-Ni films in an as-deposited state.

III. RESULTS AND DISCUSSION

Figure 1 shows the coercivity and the Fe content in the electrodeposited Fe-Ni films as functions of the content of tartaric acid in the plating bath. The Fe content shows a tendency to increase from 18 to 45 at.% with increasing content of tartaric acid. The coercivity shows a dramatic decrease from 400 to 20 A/m with increasing tartaric acid content from 0 to 15 g/L. In the region of higher acid concentrations, the Fe content and the coercivity were both saturated to 45 at.% and 60 A/m, respectively. In our previous results for the citric-acid-based bath, the same tendencies were observed, and the dramatic decrease in the coercivity could be explained by the change of the composition in the film [4]. In order to clarify the effect of Fe content in the films prepared from a tartaric-acid-based bath on the coercivity, we replotted the coercivity results in Fig. 2 as a function of the Fe content in the film. The results from our previous study for the films prepared from a citric-acid-based bath [4] are also

shown in Fig. 2 for comparison.

From the plots in Fig. 2, low coercivity values are clearly obtained at around 22 at.% Fe, and the overall trend is independent of the kind of acid. Since both the magnetocrystalline anisotropy and the magnetostriction constants in the Fe-Ni binary system become nearly zero at $\text{Fe}_{22}\text{Ni}_{78}$, the lowest coercivity of our films at around 22 at.% Fe is readily attributable to the small intrinsic magnetic and magneto-elastic anisotropy values. The change in the coercivity induced by the tartaric-acid content in the plating bath could be explained by the composition of the films.

Figure 3 shows XRD patterns of the films prepared at various concentrations of tartaric acid. It is clear from these diffraction patterns that line broadening and a shift of the diffraction angle to the lower angle side take place when the acid concentration is high. The shift of the diffraction angle indicates a change in the lattice parameter of the Fe-Ni lattice. The line broadening indicates a reduction in the grain size. These tendencies also agree with the results for the citric-acid-based bath [4].

The cathode efficiency is one of the important parameters for the electrodeposition process. Thus, we evaluated the cathode efficiency by comparing the experimental and the theoretical film masses; the cathode efficiency is defined as the actual weight of electrodeposited Fe-Ni film divided by the theoretical weight as per Faraday's law. Figure 4 shows the cathode efficiency as a function of the tartaric acid content in the plating bath. The results for citric acid [4] are also shown in Fig. 4. The cathode efficiencies for both the plating baths decrease from approximately 80 to 20 % with increasing concentration of the acids. Figure 5 shows the cathode efficiency as a function of the Fe content in the films. Cathode efficiency clearly shows a decrease with increasing Fe content, and the cathode efficiency for the tartaric-acid-based bath shows a value slightly higher than for the citric-acid-based bath at the same Fe content. This result indicates that the tartaric-acid-based bath is more effective than the citric-acid-based bath to obtain Fe-Ni films with good soft magnetic properties. From these results, we conclude that the tartaric-acid-based bath is an environmentally-friendlier plating bath and is more suitable for mass-production of Fe-Ni films as compared with the citric-acid-based bath.

IV. CONCLUSIONS

We plated Fe-Ni films from a tartaric-acid-based bath and compared the structural and the magnetic properties of the films with the results for the citric-acid-based bath. Our results are summarized as follows:

- (1) Fe-Ni films with a low coercivity of less than 30 A/m could be obtained at an Fe content of around 22 at.%.
- (2) The films Fe content was increased from approximately 18 at.% to 45 at.% with increasing content of tartaric acid in the bath.
- (3) The cathode efficiency was reduced from 80% to 20% with increasing content of tartaric acid.
- (4) The cathode efficacy for the tartaric-acid-based bath was slightly higher than that for the citric-acid-based one, making tartaric acid advantageous for massproduction.

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Figure Captions.

Fig. 1. Coercivity and Fe content in electrodeposited Fe-Ni films as functions of the tartaric acid content in the plating bath.

Fig. 2. Relationship between the coercivity and the Fe content (replot of the results in Fig. 1). The results for citric acid from Ref. 4 are also shown.

Fig. 3. XRD patterns of the films prepared with various contents of tartaric acid.

Fig. 4. Cathode efficiency as a function of the tartaric acid content in the plating bath. The results for citric acid are also shown.

Fig. 5. Relationship between the cathode efficiency and the Fe content.

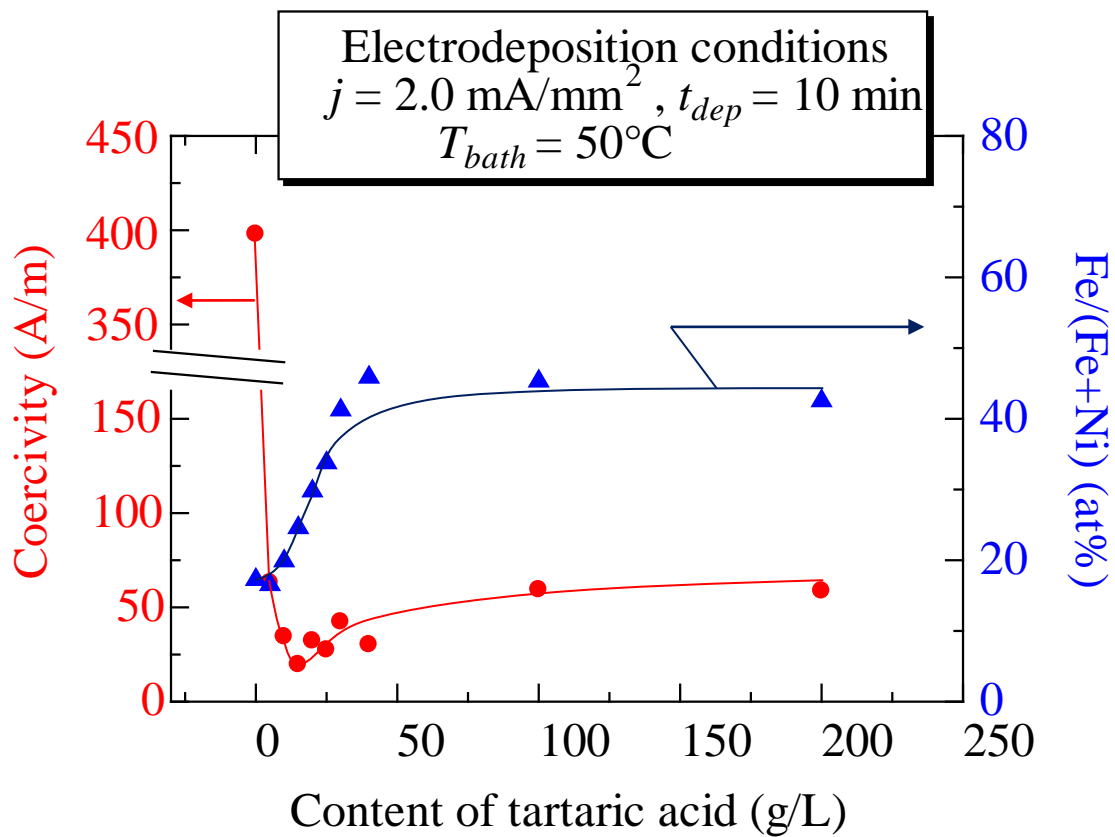


Fig. 1.

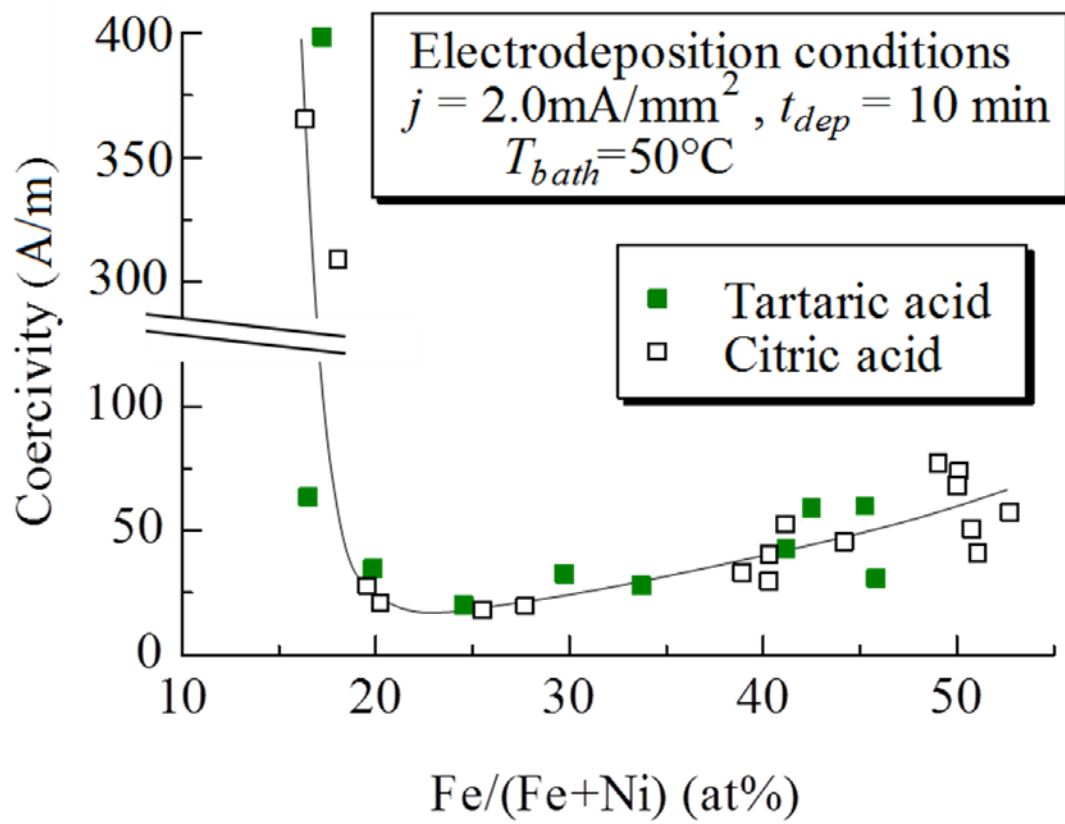


Fig. 2

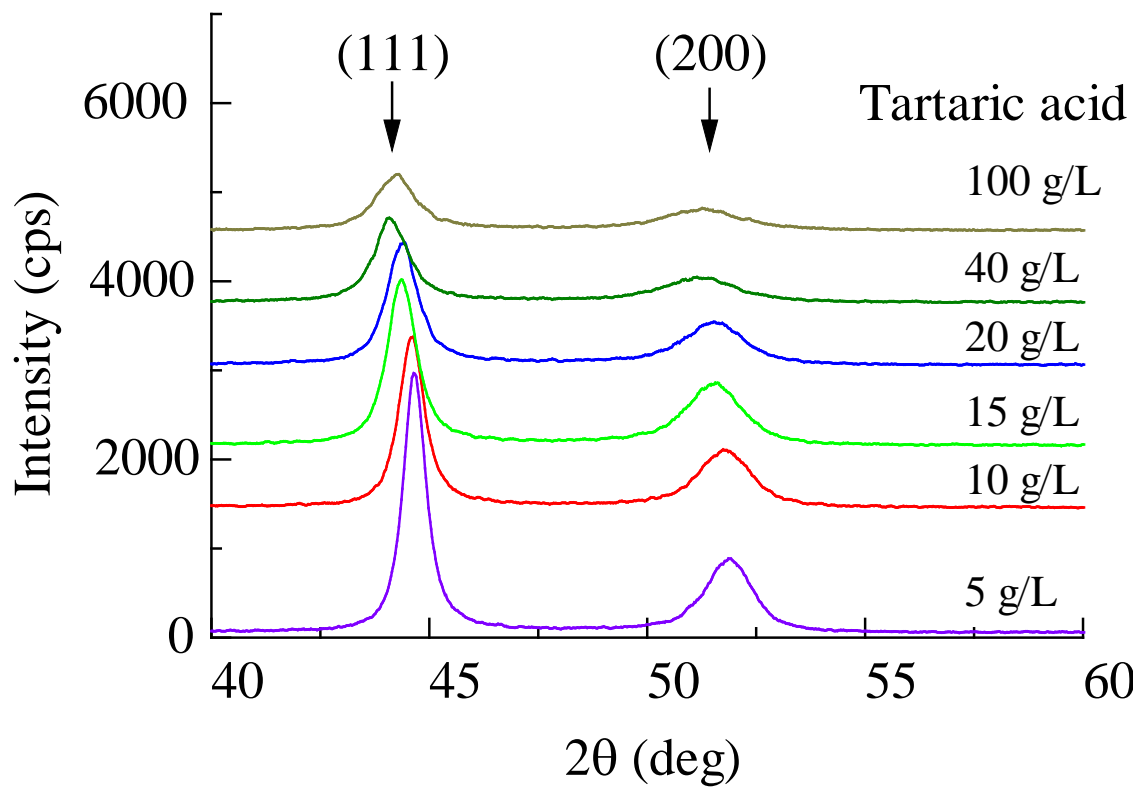


Fig. 3

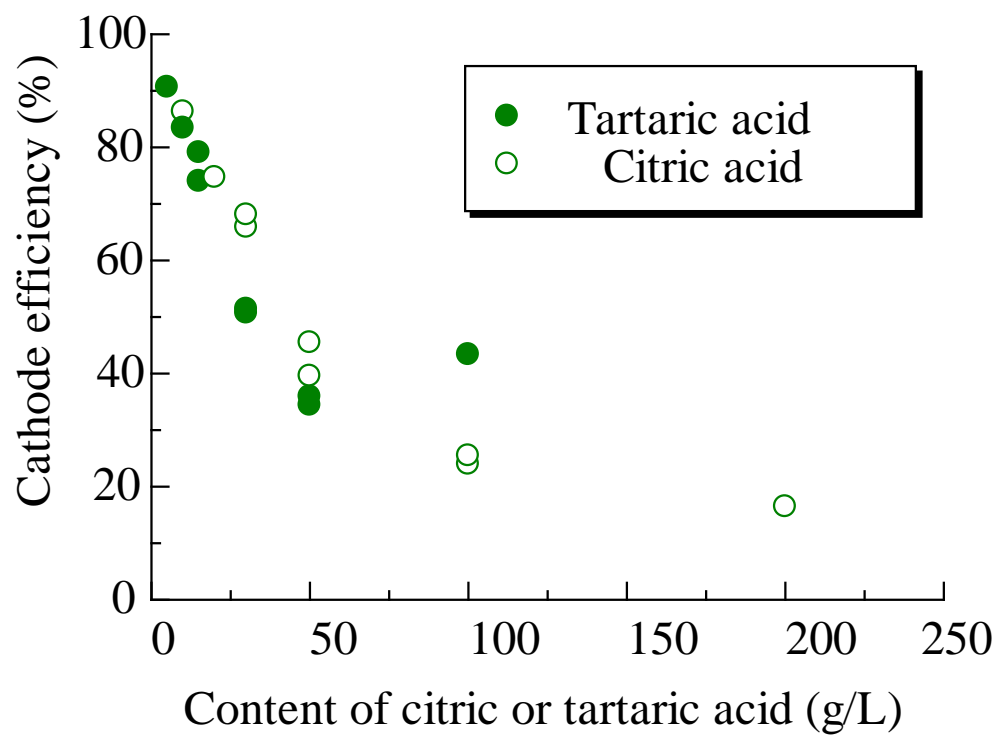


Fig. 4

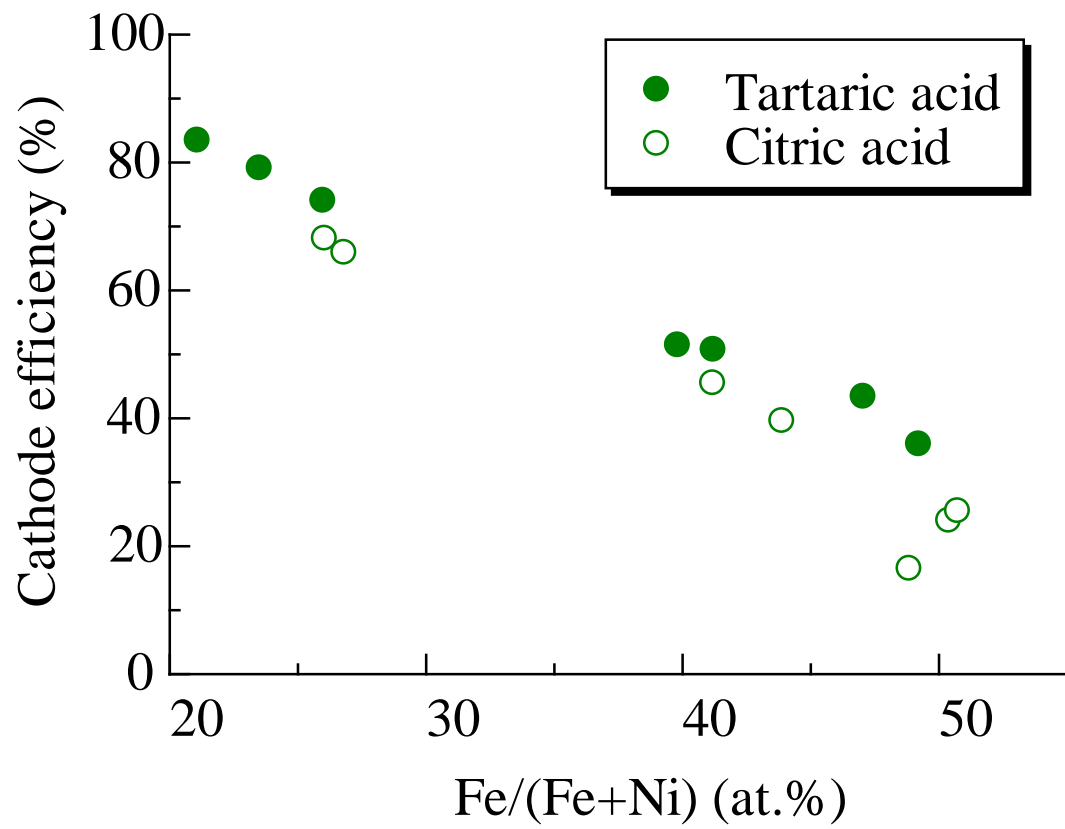


Fig. 5