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Preparation and influence of pH on the dynamic magnetic property of magnetic FeCoC films

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HIGHLIGHTS

- We have successfully prepared FeCoC soft magnetic films by electrochemical deposition method.
- The resonance frequency can be controlled by changing pH value.
- A widely absorption peak will be obtained when the pH value is appropriate.

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ABSTRACT

FeCoC films were successfully prepared by electrochemical deposition method in different citric acid concentrations and pH values. The morphology, structure and magnetic properties were investigated. FeCoC films deposited at different citric acid concentrations have good soft magnetic performance. As the pH value increases from 2.49 to 6.02, the atomic ratio of Fe:Co range from 0.72 to 0.95. The coercivities of the films deposited at different pH values first increase and then decrease with increasing pH. The resonance frequency of the films can be tuned by controlling the pH value, and in an appropriate pH value a wide absorption peak can be obtained.

1. Introduction

Recently, soft magnetic films have attracted much attention due to their potential applications, such as magnetic recording heads [1], magnetic sensors [2] and microwave noise filters [3]. For their high frequency applications, high saturation magnetization ($4\pi M_s$) and remarkable in-plane uniaxial magnetic anisotropy (H_k) are important [4–8]. FeCo thin films have received much attention because of their high saturation magnetization [9,10]. However, the magnetic properties of pure FeCo film cannot meet the demand for high frequency applications. Because on one hand, the large magnetocrystalline anisotropy and magnetostriction of FeCo than soft FeNi films that limit their high frequency applications [11]. On the other hand, the eddy current loss is serious because of the lower resistivity of metal films [12]. In recent years, many reports were devoted to enhance the soft magnetic properties of FeCo film by various methods [13–15]. One way to obtain excellent soft magnetic properties is to add the third element (like B, P, N, C, Zr, Cd) into FeCo films [16–18]. Therefore many researchers were committed to study the magnetic properties of FeCo-based alloy, especially high frequency, i.e., dynamic magnetic properties [17–20]. Among those alloy, FeCoC alloy are expected to be one of the best soft magnetic films with excellent high frequency performance [21]. Also, the excellent static magnetic properties of FeCoC films prepared by sputtering have been demonstrated by Liu et al. [22]. However, its dynamic magnetic properties were not mentioned.

Up to now, molecular beam epitaxy (MBE) [23], sputtering [24], chemical plating [25], electrochemical deposition methods [26]

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have been used to fabricate magnetic films. Compared with other methods, electrochemical deposition method has a broad range of applications due to its low cost, high efficiency and easy operation. Moreover, it also can be used to prepare large-area films via regulating various experimental conditions. So far, however, few reports [27] have shown that FeCoC films were prepared by electrochemical deposition method. In addition, the morphology and microstructure of the film are closely related to deposition potential, deposition temperature, additives and electrolyte pH value in electrochemical deposition process. Among those parameters, particularly, the pH value of electrolyte is an important factor for influencing the properties of the soft magnetic films [28].

In this work, magnetic FeCoC films were prepared by electrochemical deposition method. We used the citric acid as the C source to append C element into the FeCo films. According to the XPS images, the C element has been successfully incorporated into the FeCo films, and the FeCoC films show good soft magnetic properties. Furthermore, the effect of pH on the dynamic magnetic property was also studied.

2. Experimental

FeCoC magnetic films were fabricated by electrochemical deposition method, which is based on the redox reaction principle. In the experiment, we used a stationary three-electrode (work electrode, anode electrode and reference electrode) cell, which is controlled by potentionstatic mode. Work electrode is a 2 cm × 2.5 cm ITO conductive glass, anode electrode is a 1.5 cm × 1.5 cm platinum plate and reference electrode is a saturated calomel electrode (SEC). The resultant FeCoC films were deposited on the work electrode. Before the experiment, the ITO conductive glasses were orderly cleaned in acetone and alcohol each for 20 min by ultrasonics and then washed using distilled water. All the depositions were performed at 40 °C electrolyte temperature. Meanwhile, a 800 Oe magnetic field was applied parallel to the ITO conductive glass in order to induce uniaxial anisotropy in the film plane. The electrolyte consists of 0.05 mol/L $FeSO_4 \cdot 7H_2O$, 0.05 mol/L $CoSO_4 \cdot 7H_2O$, 1 g/L saccharine, 1 g/L glycine, 1 g/L ascorbic acid, and 0.4 mol/L H_3BO_3. The deposition potential was −1.3 V and deposition time was 500 s. Citric acid was used as the C source. FeCoC films were fabricated at 0, 0.005, 0.008, 0.01, 0.015, 0.02 mol/L citric acid concentration as shown in Table 1. Then, we investigated the influence of pH value on the FeCoC films when the citric acid concentration is 0.01 mol/L. The FeCoC films were deposited at pH 2.49, 2.53, 2.60, 3.17, 3.59 and 6.02, which is controlled by using NH_4Cl and NaOH. The composition and operating conditions for electrochemical deposition were shown in Table 1.

The crystalline structure of FeCoC alloy films was characterized by X-ray diffraction which is using Cu-K_α radiation (XRD, PANalytical X′ pert Pro, Holland). The elements composition was analyzed by X-ray photoelectron spectroscopy (XPS) and Energy dispersive X-Ray spectroscopy (EDS). The morphology and thickness of the samples were observed by the scanning electron microscopy (SEM, Hitachi S-4800, Japan). Vibrating sample magnetometer (VSM, Lakeshore 7304, USA) was used to measure the static magnetic properties and vector network analyzer (VNA, Agilent E8363B, USA) was used to measure the permeability spectra by shorted microstrip transmission-line method from 100 MHz to 8 GHz [29].

Table 1
Compositions and operating conditions for electrochemical deposition FeCoC films.

Chemical composition	Concentration
$FeSO_4 \cdot 7H_2O$	0.05 mol/L
$CoSO_4 \cdot 7H_2O$	0.05 mol/L
Citric acid	0–0.02 mol/L
pH	2.49–6.02
Saccharine	1 g/L
Glycine	1 g/L
Ascorbic acid	1 g/L
H_3BO_3	0.4 mol/L
Bath temperature	40 °C
Operating time	500 s
Deposition potential	−1.3 V vs. SCE

3. Results and discussion

3.1. Effect of the citric acid concentration on microstructure and magnetic properties of FeCoC films

The XRD patterns for FeCoC films deposited at different citric acid concentrations are shown in Fig. 1(a). It can be seen clearly that when the films deposited in less than 0.005 mol/L citric acid concentration exhibit bcc and fcc phase of FeCo. The duplex structures of FeCo gradually turn into the fcc phase when the films deposited in above 0.008 mol/L citric acid concentration. There is no FeC or CoC peak in the XRD patterns, thus it reveals that the C element may be doped into the interstice of the FeCo crystal lattice.

For the purpose of verifying whether the C element is doped or not. XPS measurement was executed. Before measuring, instrument calibration is implemented by C element, therefore a small number of the C element may remain in the testing system and the

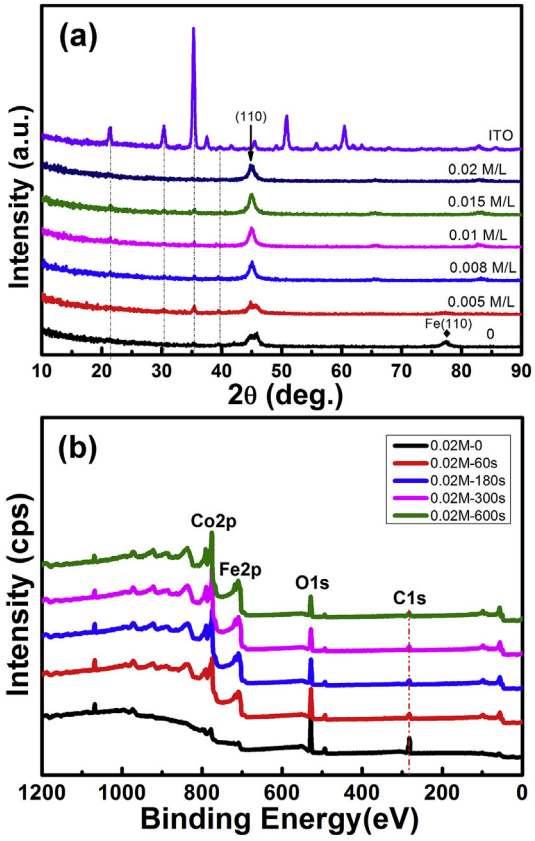

Fig. 1. (a) XRD patterns for the FeCoC magnetic films deposited at different citric acid concentrations and (b) XPS spectra for FeCoC magnetic films deposited at 0.02 mol/L citric acid concentration etching at different times.