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Preparation and magnetic properties of iron titanium oxide nanotube arrays

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Abstract

FeTi alloy was prepared by a vacuum smelting method, iron titanium oxide nanotube arrays have been made directly by anodization of the FeTi alloy. Morphologies and microstructures of the samples were characterized by scanning electron microscope, transmission electron microscope, and X-ray diffractometer. Influences of temperature and H_2O concentration on the morphologies of the nanotube arrays have been discussed in detail. Magnetic properties of the samples have also been investigated. The as-prepared samples were amorphous. When annealed at 500 °C and 550 °C, pesudobrookite Fe_2TiO_5 was obtained. At 600 °C, there were mixed Fe_2TiO_5, rutile TiO_2, and α-Fe_2O_3. Magnetic performance of the nanotube arrays exhibited high sensitivity to temperature and changed interestingly upon annealing. The values of the coercivity and remanence were 340 Oe and 0.061 emu/g respectively for the sample annealed at 550 °C.

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1. Introduction

That the properties of materials depend on their morphologies is well documented [1,2]. Nanotube arrays are of great interest because they exhibit enhanced or new properties compared to other morphologies. Many kinds of oxide nanotube arrays (TiO_2, ZnO, ZrO_2, Fe_2O_3, HfO_2, Ta_2O_5, Eu_2O_3, etc.) have been prepared and excellent properties attained [3–10]. For example, the ultrathin α-Fe_2O_3 nanotube arrays showed excellent photoactivity and an improved charge transport ability which was observed to be 40 to 50 times higher than those of the nanoparticles [8]. The TiO_2 nanotube arrays showed significantly higher charge-collection efficiencies and light-harvesting efficiencies than those of the nanoparticles [11].

Compared to simplex Fe_2O_3 and TiO_2, iron–titanium composite oxides showed good stability and ferrimagnetic–paramagnetic behavior at room temperature [12,13]. To date, iron titanium oxide particles, thin films, or hollow spheres have been synthesized by solid-state reactions, co-precipitation, hydrothermal, or sol–gel methods and studies show that iron titanium oxide materials have the potential to be applied as non-linear optical devices, catalysts, Li-ion batteries, electrodes for the electrolysis of water, etc. [14–21]. However, to the best of our knowledge, there are few studies on the preparation of iron titanium oxide nanotube arrays [22,23]. For this research, iron titanium oxide nanotube arrays were prepared by the anodization of FeTi alloy for the first time and their magnetic properties were discussed in detail.

2. Experimental details

The FeTi alloys used in this study were obtained by smelting mixed iron and titanium (99.9% purity, mole ratio 1:1) in an arc melting furnace. Before the experiment, FeTi alloys were cut into 20 mm × 10 mm × 2 mm pieces and polished by means of metallographic abrasive papers, cleaned in ethanol and deionized water with ultrasonic washer, finally dried in air. The electrochemical set-up used in this work consisted of a two-electrode arrangement with FeTi alloy as the anode and the platinum (20 mm × 20 mm × 0.1 mm) as the cathode. The distance between the anodic and cathodic electrodes was kept at 20 mm. Electrolytes used in this

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anodization process were ethylene glycol (EG) containing small amount of NH_4F and H_2O. FeTi alloys were anodized at a constant temperature and voltage for 3 h, during the anodization processes, the solutions were stirred by a magnetic stirrer. When the anodization process was finished, the samples were rinsed with deionized water and air-dried. The oxide layer was then peeled off from the substrate, calcinated, and characterized. Morphologies and structures of the samples were investigated using a scanning electron microscope (Hitachi, S-4800), transmission electron microscope (JEOL, 200CX), and X-ray diffractometer (Rigaku, D/MAX-2500) respectively. Magnetic properties of the samples were investigated using a vibrating sample magnetometer (Lake Shore, 7304).

3. Results and discussion

It is well known that the formation of nanotubes is strongly dependent on the anodization conditions including: temperature, voltage, water content, and so forth. Previous studies showed that titania nanotube arrays and iron oxide nanotube arrays, etc. can be prepared in EG electrolyte [23–26]. Thus, the EG electrolyte was chosen to prepare iron titanium oxide nanotube arrays and a set of anodization experiments were carried out to explore the appropriate conditions. Results showed that the influences of temperature and water content on the morphology and structure of the iron titanium oxides were remarkable.

Figs. 1 and 2 show the morphologies of the samples after anodization in EG electrolyte containing 0.25 wt% NH_4F and 2 wt% H_2O at different temperatures. From the SEM images it was evident that a nanoporous oxide layer was formed at 20 °C, the pore diameters of the oxide layer were irregular (Fig. 1(a)). The cross-sectional view of the oxide layer was rather compact and rough with a thickness of around 2 μm (Fig. 1(c)). The mushroom-like bottoms were irregular and uneven with a diameter of approx. 75 nm (Fig. 1(e)). When the temperature increased to 30 °C, nanotubular structures have been formed, the nanotube diameter was approx. 70 nm (Fig. 1(b)). The cross-sectional view of the oxide layer still

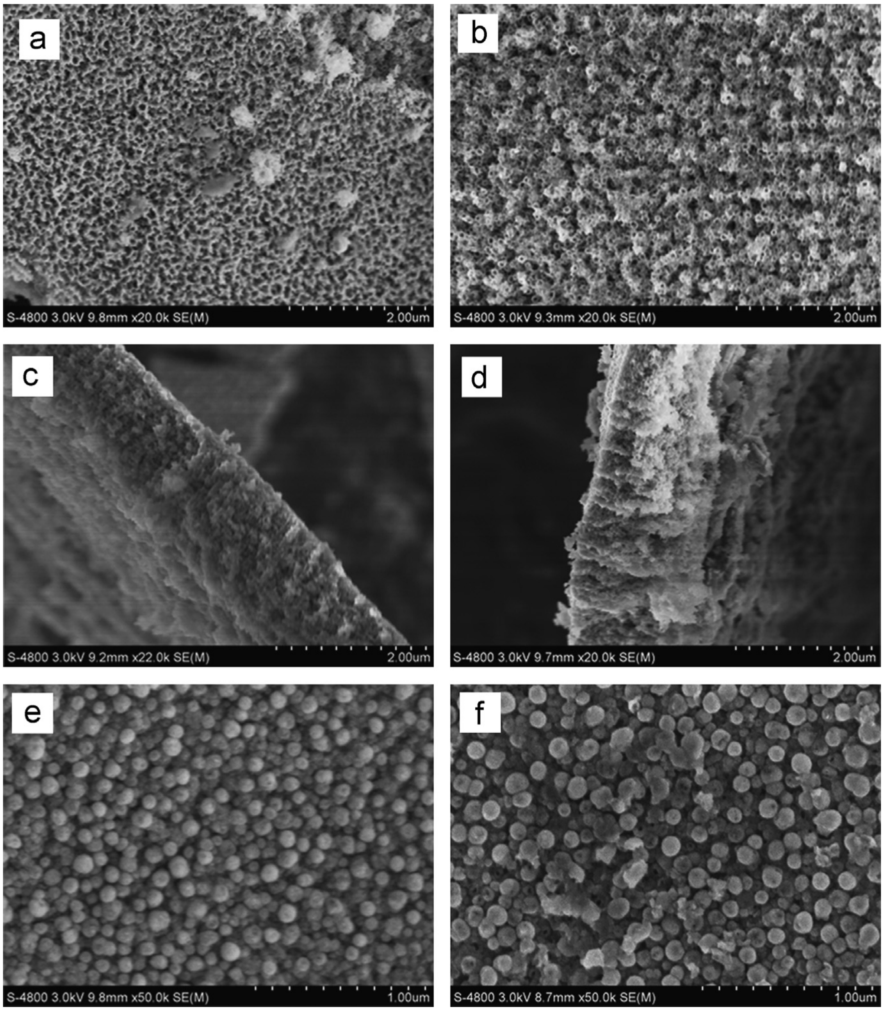

Fig. 1. SEM images of samples anodized in EG electrolytes containing 0.25 wt% NH_4F and 2 wt% H_2O for 3 h at 40 V. (a) 20 °C; (b) 30 °C; (c) cross-sectional view of (a); (d) cross-sectional view of (b); (e) bottom view of (a); and (f) bottom view of (b).

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