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RESEARCH ARTICLE

Synthesis and Characterization of Fe-Ag Alloy by Pulsed Electrodeposition†

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Abstract: An alloy of the immiscible constituents iron and silver was prepared by pulsed electrodeposition method from a single bath containing ferric nitrate, silver nitrate and sodium perchlorate. The surface morphology of the as deposited alloys was investigated by using field emission scanning electron microscopy (FESEM) analysis while the crystal structure was examined by X-ray diffraction analysis. XRD analysis showed that alloy exhibits *fcc* structure. FESEM analysis depicted the formation of dendrites over the spherical particles. The vibrating sample magnetometer (VSM) study unveiled that the Fe-Ag alloy deposits were ferromagnetic in nature and Curie temperature was found to be 585.4 °C.

Keywords: Fe-Ag alloy, ferromagnetic alloy, Pulsed electrodeposition

Introduction

Magnetic alloys prepared from groups of magnetic and non magnetic materials plays an important role in material science due to their enhanced physical properties, such as giant magnetoresistance, superparamagnetism. Moreover, they have important technological applications in magnetic recording, in optical devices and in sensors[1]. Fe-Ag alloy is such a two-component alloy system consisting of magnetic iron and nonmagnetic silver metal.

Iron can form alloys or solid solutions with almost any metal, while there are a few metallic elements with which iron is immiscible. Ag is one such example metal. Their solubility in both liquid and solid state is less than 1%[2]. However, in recent years, it has been shown that metastable and homogeneous alloys of Fe–Ag system can be formed by using many special techniques[3-5]. Depending on the preparation technique these metastable alloys are stable up to few hundred degree above room temperature. Among them electrodeposition has several advantages over other methods as it is simple to assemble and operate, is quite inexpensive.

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This alloying process is difficult because the difference in standard electrode potentials of silver (0.779V) and iron (-0.440V) metals is considerably large. Appropriate complexing agents can be used to reduce the difference in their standard electrode potential. There are only few studies exist on this method in literature. In this work Fe/Ag alloy has been prepared by electrodeposition method. In the present work Pulsed Electro Deposition (PED) method was employed to form the alloy.

Experimental

Fe-Ag alloy deposit was obtained using stainless steel plate as cathode. Plating is carried out in a perchlorate medium. The complexing agents are used to reduce the standard electrode potential for simultaneous deposition of silver and iron. The electrolytes were prepared using double distilled water and the pH of the electrolyte solution is adjusted to 3 adding a small amount of perchloric acid. The current density was kept at 0.2A/cm$^{2.}$ The electrolyte contains the aqueous solutions of ferric nitrate (0.1 M), silver nitrate (0.01 M) and sodium perchlorate (0.2 M). It also contains the complexing agents like boric acid (0.3 M) and sodium gulconate (0.2 M). These operating conditions and the electrolytic bath composition were optimized after performing a number of trials to obtain a homogeneous deposit of the alloy.

After deposition the deposits were rinsed 4 to 5 times with distilled water and finally washed with acetone and dried. Alloy and as prepared sample was characterized for their elemental composition, crystal structure, magnetic behavior and surface morphology. These studies were carried out by using the FESEM equipped with energy dispersive x-ray spectrometer (EDX) (SU6600, Hitachi), Inductively coupled plasma optical emission spectroscopy (ICP-OES) analysis using Perkin Elmer Optima 5300 DV Inductively Coupled Plasma (ICP), X-ray diffractometer (GE- X-Ray Diffraction system - XRD 3003 TT) with Cu Kα (λ=0.15406 nm) and Vibrating Sample Magnetometer (VSM, EG&G PARC, Model 4500).

Results and Discussion

Compositional analysis

The chemical composition of Fe-Ag alloy deposited at current densities 0.2A/cm^2 was determined by ICP-OES. Atomic percentage of silver-iron alloy sample was found to be 56.02:43.98. The energy dispersive X-ray analysis spectrum of Fe-Ag alloy deposited at 0.2 A/cm^2 also confirms the presence of silver and iron in the sample.

XRD analysis

The structural analysis was carried out using XRD. The XRD patterns of the pulsed electrodeposited alloy are shown in the Figure 1.

These patterns exhibit reflections from (111), (200), (220), (311) and (322) planes indicating the *fcc* structure which is similar to the Ag *fcc* structure as per JCPDS file card No: 89-3722. Though the presence of iron in the deposits is ascertained by ICP-OES, EDAX and the magnetic studies, the peaks corresponding to the *fcc* silver phase are only detected. Peaks corresponding to neither metallic iron nor iron oxide are observed. These results agree with those reported by Roy *et al*[6]. The non observance of iron peaks in the alloy which has about 44% of iron, it may be due to incorporation of iron atoms into the silver lattice, since the atomic radius of iron (0.126 nm) is smaller than that of silver (0.144 nm).

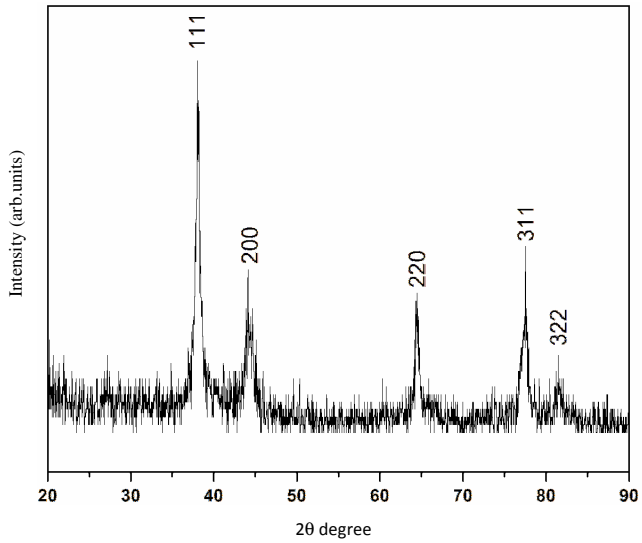
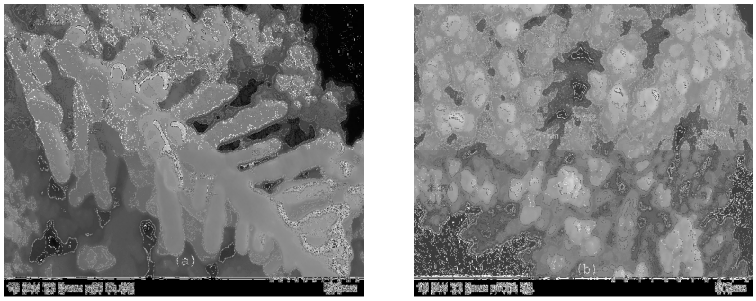


Figure 1. XRD pattern of Fe-Ag alloy

Surface morphology analysis

The surface morphology of the synthesized Fe-Ag alloy at 0.2 A/cm^2 is analyzed using FESEM and these images are shown in Figure 2(a-b).

Figure 2(a) shows the growth of dendrites over the spherical particles with size of about 20-40 nm and the magnified image of the dendrites shown in Figure 2(b) clearly illustrates that the dendrite comprises of small spherical particles. This is due to the PED process which facilitates fresh nucleation instead of the already existing particles growing bigger in size.



a b

Figure 2. FESEM images of Fe-Ag alloy deposited at 0.2 A/cm^2

Magnetic studies

Hysteresis loops of the samples were obtained using a vibrating sample magnetometer with a maximum applied field of 7000 Oe at room temperature to study the magnetic property.

Figure 3 shows the magnetization curve of the pulsed electrodeposited Fe-Ag alloy. The hysteresis behavior observed for the Fe-Ag alloy indicates its soft ferromagnetic nature. The saturation magnetization (M_s) value is 9.1842 emu/g calculated from the loop.

The Curie temperature (T_c) was estimated for Fe-Ag using thermo magnetic studies. Figure 4 shows the temperature *vs.* magnetization graph.

The apparent drop in temperature at 585.4 ^{0}C is attributed to Curie point (T_c) of electrodeposited Fe-Ag alloy. The one step Curie transition signifies the formation of the alloy with the single phase which supports the XRD result. These is in agreement with the Tc (570 ^{0}C) reported by Liu *et al*[7]. The Curie transition temperature of iron in the deposit is seems to be lowered to 585.4 ^{0}C from the curie temperature of iron (770 ^{0}C). This is due to formation of Fe-Ag alloy with incorporation of the iron atom in the silver lattice.

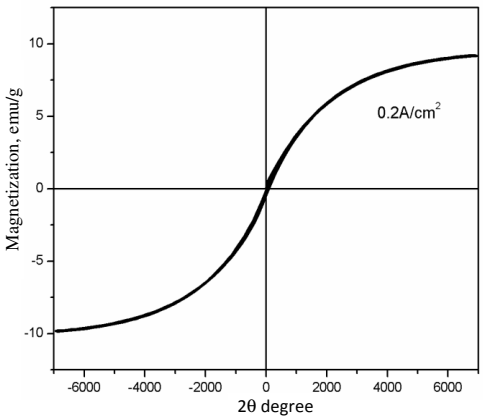


Figure 3. Hystesis loops of Fe-Ag alloy deposited at 0.2A/cm^2

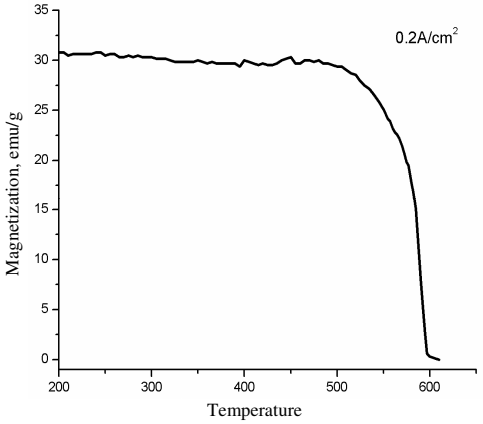


Figure 4. Shows the transition temperature of Fe-Ag alloy

Conclusion

The immiscible system of iron and silver with a large difference in their deposition potential has been synthesized by pulsed electrodeposition from a complex electrolyte. XRD analysis shows that alloy exhibits fcc structure. FESEM analysis reveals that in the Fe-Ag alloy deposits the spherical nano sized particles grow to form dendrites. The vibrating sample magnetometer (VSM) study reveals that the Fe-Ag alloy deposit was with ferromagnetic activity and Curie temperature is found to be 585.4 ^{0}C

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