

Electrochemical Behaviours of La-Fe Alloys Electrodeposited in DMSO Solution

GUO LIN and YIN WEI*

Department of Chemistry, Guangdong Education Institute, Guangzhou 510303, P.R. China

E-mail: yinwei@gdei.edu.cn

The brown metallic luster La-Fe alloy powders were prepared by potentiostatic electrolysis technique with a cathode of fine copper threads in DMSO solution at room temperature. The La-Fe alloy powders containing 28.40-43.54 % lanthanum with the changes of concentration of LaCl_3 . DMSO solutions show that the different weight of rare earth alloy powders can be obtained by controlling the concentration of lanthanum solution. The size of metal grains is about 0.1 μm to 20 μm . The turning potentials of -2.41, -1.71 and -1.17 V are attributed to reduction of La^{3+} , Fe^{2+} and Fe^{3+} ion, respectively. The turning potentials of -3.40 and -1.46 V are assigned to the oxidation of La and Fe metals, respectively.

Key Words: Alloy, Lanthanum, Electrodeposition, DMSO.

INTRODUCTION

The alloys of lanthanides and ferrous metal have an excellent magnetic characteristic, applied in many fields, including metallurgy industry, agricultural machine, and magnetic therapy and so on. It is significant and urgent to investigate electrochemical behaviours of various lanthanide alloys, due to widely applied in electrochemical field. The preparation of electrochemical method is one of important ways for rare earth alloys. The melting electrolysis methods of rare earths alloys are rigorous to equipments and the rare earth alloys are difficult to deposit in aqueous solution by electrochemical methods. The literatures¹⁻⁴ reported that the alloy films of rare earth can be obtained by electrodeposition in organic solutions at room temperature. This paper reports the preparation of La-Fe alloy powders were prepared by electrochemical method with a cathode of fine copper threads in DMSO solution at room temperature. The electrochemical behaviours of the La-Fe alloys were investigated with cyclic voltage-current analysis.

EXPERIMENTAL

LaCl_3 was obtained by the reaction of La_2O_3 (99.99 %) and conc. hydrochloric acid (AR) and dehydrated in vacuum at 393 K, FeCl_2 and LiCl were dehydrated in vacuum at 453 K. Dimethylsulfoxide (DMSO) was dehydrated with 4 Å molecular sieves and distilled at reduced pressure to remove impurities^{5,6}. A simple two-electrode cell was used to prepare powders of La-Fe alloy. A cathode of fine copper

threads ($\text{Cu} = 99.9\%$, $\Phi = 0.015\text{ cm}$) was used as working electrodes and a platinum foil (0.24 cm^2) was used as the counter electrode. All electrochemical experiments were carried out in nitrogen atmosphere at room temperature. MYJ—G_A direct current potentiostatic electric source was used to electrolyses and potential determined values was applied. The X-ray powder diffraction measurements of samples were carried out on a Rigaku D/max-3A diffractometer using $\text{CuK}\alpha$ radiation. The morphologies and components of the alloy powders were analyzed with a S-520/ISIS-300 scanning electron microscopy/energy dispersive X-ray spectrometer. The electrodeposited powders of La-Fe alloy were pressed to piece connected with a platinum wire and used as working electrode. A platinum foil (0.24 cm^2) was the counter electrode and a saturated calomel electrode (SCE) was the reference electrode with a salt bridge. The LK98 electrochemical integrated testing instrument was employed to complete the electrochemical analysis.

RESULTS AND DISCUSSION

Preparation of La-Fe alloy powders by potential-static technique: La-Fe alloy powders were prepared by potentostatic electrolysis technique in two kinds of electrolyte system: A - $0.033\text{ mol L}^{-1}\text{ LaCl}_3$ - $0.1\text{ mol L}^{-1}\text{ FeCl}_2$ - $0.2\text{ mol L}^{-1}\text{ LiCl}$ (supporting electrolyte)-DMSO; B - $0.050\text{ mol L}^{-1}\text{ LaCl}_3$ - $0.1\text{ mol L}^{-1}\text{ FeCl}_2$ - $0.2\text{ mol L}^{-1}\text{ LiCl}$ (supporting electrolyte)-DMSO. A Pt foil (0.24 cm^2) was used as counter electrode. To codeposit La-Fe alloy powders employed the potentials from 30 to 45 V using MYJ—G_A direct current potentiostatic source electrolyzed for 1 h.

The images of the La-Fe alloy powders show that La-Fe alloy powders obtained were brown, separated, uniform and metallic luster and the size of metal grains is about 0.1 to 20 μm (Fig. 1). The content of La in alloy powders is from 28.40 to 43.54 % in different electrolyte systems tested by energy dispersive X-ray spectrometer

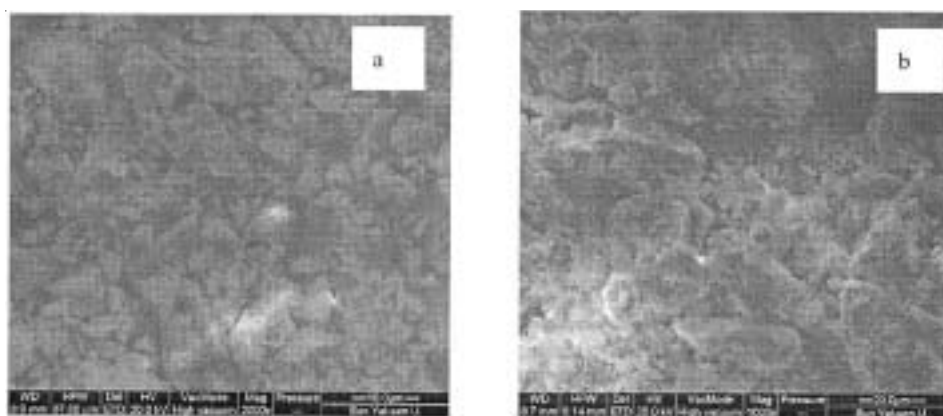


Fig. 1. The morphologies of La-Fe alloy powders electrodeposited in different DMSO solutions
a = $0.033\text{ mol L}^{-1}\text{ LaCl}_3$ - $0.1\text{ mol L}^{-1}\text{ FeCl}_2$ - $0.2\text{ mol L}^{-1}\text{ LiCl}$ -DMSO
b = $0.050\text{ mol L}^{-1}\text{ LaCl}_3$ - $0.1\text{ mol L}^{-1}\text{ FeCl}_2$ - $0.2\text{ mol L}^{-1}\text{ LiCl}$ -DMSO

(Fig. 2). The energy dispersive X-ray spectra exhibits the components of the alloy selected area and the content of La is 21.63 %, closed to the theory value. The morphology of La-Fe alloy powders shows that the maximum size of the alloy mass is about 20 μm (Fig. 3). The experimental results show that the micrometer materials of rare earth alloys can be obtained by controlling electric current of electrodeposition and the different content of rare earth in alloys powders can be obtained by controlling concentration of solution.

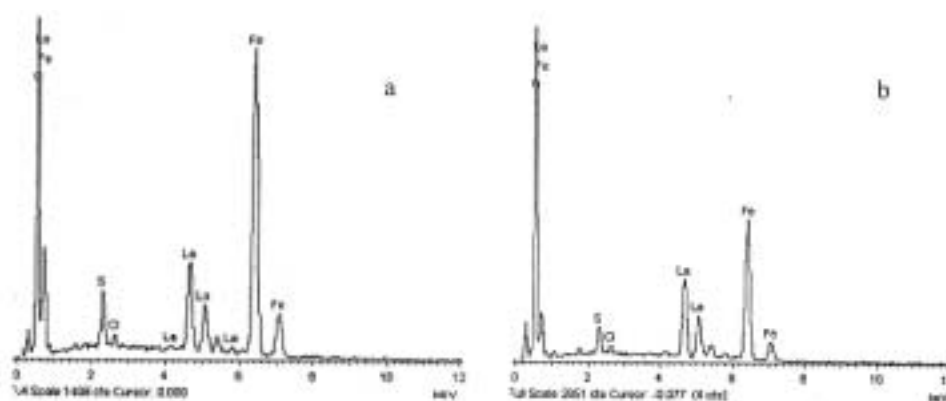


Fig. 2. Energy dispersive X-ray spectra of La-Fe alloy powders
 a = 0.033 mol L⁻¹ LaCl₃-0.1 mol L⁻¹ FeCl₂-0.2 mol L⁻¹ LiCl-DMSO,
 b = 0.050 mol L⁻¹ LaCl₃-0.1 mol L⁻¹ FeCl₂-0.2 mol L⁻¹ LiCl-DMSO

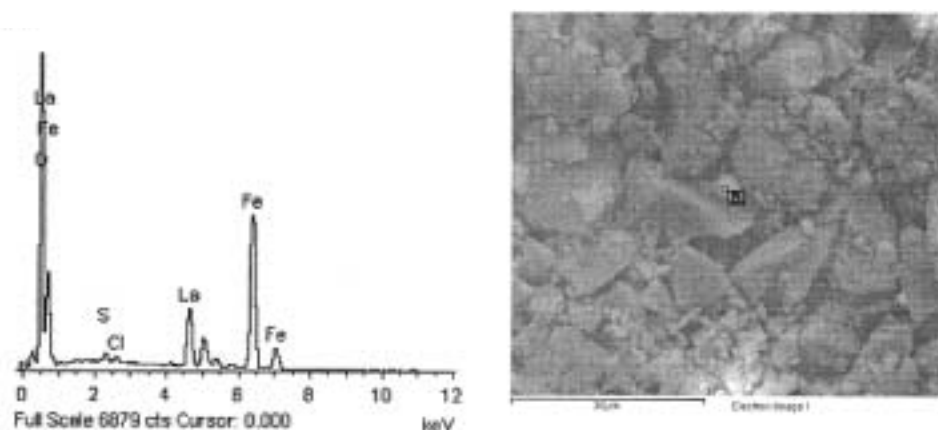


Fig. 3. Energy dispersive X-ray spectra (selected area) and the SEM of La-Fe alloy powders
 System: 0.033 mol L⁻¹ LaCl₃-0.1 mol L⁻¹ FeCl₂-0.2 mol L⁻¹ LiCl-DMSO

Electrochemical behaviours of La-Fe alloys: The piece of La-Fe alloy connected with a platinum wire from different systems separately used to cyclic voltammogram analyze in nitrogen atmosphere at 298 K by LK98 electro-chemical integrated testing

instrument. There are two anodic turning potentials at -3.40 and -1.46 V, corresponding to the dissolution of La and Fe and there are 3 cathodic turning potentials at -2.41, -1.71 and -1.17 V, attributed to the reduction of La^{3+} , Fe^{2+} and Fe^{3+} ions, respectively (Fig. 4). Fig. 5 shows that there are 2 anodic turning potentials at -3.40 and -1.51 V, corresponding to the dissolution of La and Fe and 3 cathodic turning potentials at -2.41, -1.71 and -1.17 V, correspondence to the reduction of La^{3+} , Fe^{2+} and Fe^{3+} ions. The anodic turning potentials of -3.40 and -1.46 V are attributed to dissolution of La and Fe of the alloys and the cathodic turning potentials of -2.41, -1.71 and -1.17 V are attributed to reduction of La^{3+} , Fe^{2+} and Fe^{3+} .

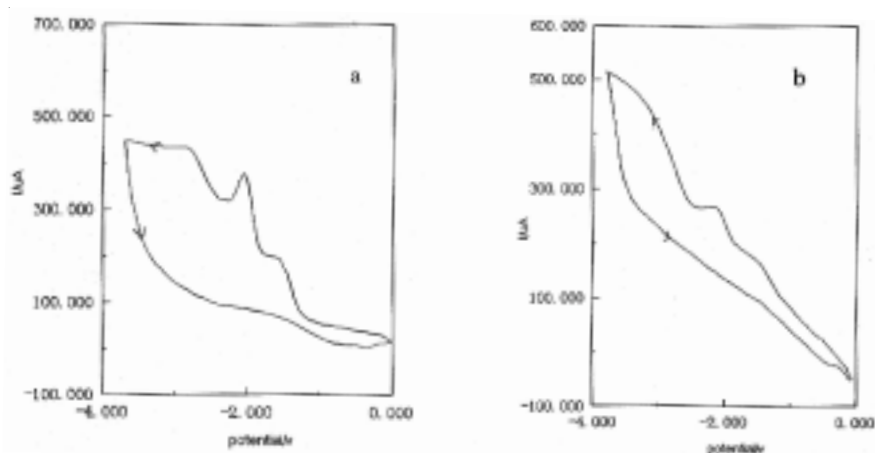


Fig. 4. Cyclic voltammograms of La-Fe alloys piece
 $0.033 \text{ mol L}^{-1} \text{ LaCl}_3$ - $0.1 \text{ mol L}^{-1} \text{ FeCl}_2$ - $0.2 \text{ mol L}^{-1} \text{ LiCl}$ -DMSO
 $T = 298 \text{ K}$, $v = 20 \text{ mv/s}$, $A = 2.0 \text{ cm}^2$, cyclic orders: a-1st, b-2nd

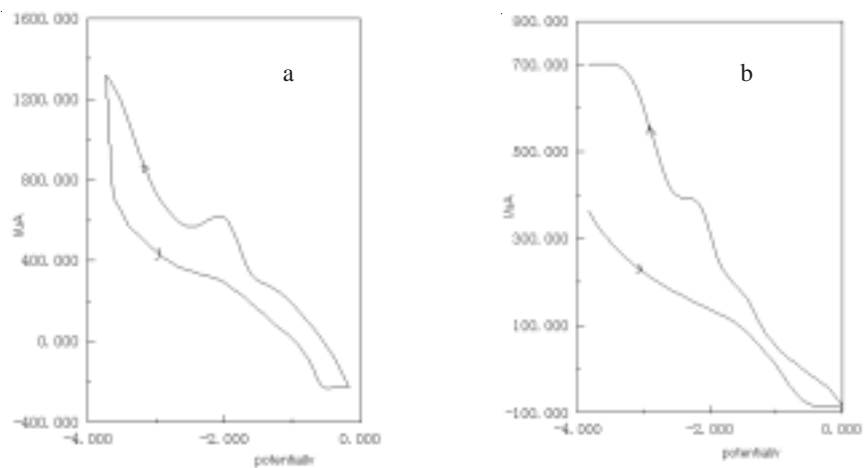


Fig. 5. Cyclic voltammograms of La-Fe alloy
 $0.050 \text{ mol L}^{-1} \text{ LaCl}_3$ - $0.1 \text{ mol L}^{-1} \text{ FeCl}_2$ - $0.2 \text{ mol L}^{-1} \text{ LiCl}$ -DMSO
 $T = 298 \text{ K}$, $v = 20 \text{ mv/s}$, $A = 1.5 \text{ cm}^2$, cyclic orders: a-1st, b-2nd

Conclusion

La-Fe alloy powders were obtained by potentiostatic electrolysis technique in DMSO solution with a cathode of fine copper threads. The morphologies of alloy powders are brown, separated, uniform and metallic luster. The sizes of metal grains are about 0.1 to 10 μm . The weight of La in La-Fe alloy powders varies from 28.40 to 43.54 % by the changes of concentration of LaCl_3 solution. It shows that different content of rare earth alloys powders can be obtained by controlling concentration of solution. The turning potentials of -2.41, -1.71 and -1.17 V are attributed to reduction of La^{3+} , Fe^{2+} and Fe^{3+} , respectively. The turning potentials of -3.40, -1.46 V are attributed to oxidation of La and Fe.

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