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Electrochemical preparation process of Mg-La alloys for solid hydrogen storage

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ABSTRACT

The objective of this work is to characterize Magnesium-Lanthanum alloy elaborated by means of an electrodeposition technique using an aqueous solution based on Magnesium chloride and Lanthanum(III) Nitrate. The Mg-La alloy is deposited on a copper cathode plate used as a cathode. Chemical composition and structure as well as the morphology are investigated respectively by EDS, XRD, FTIR and SEM.

EDS analysis shows the presence of three major elements (Mg, La and O) and a little amount of Cl. Morphological characterization reveals that heterogeneous chemical structures are formed on the surface of sample.

X-ray results showed that the obtained deposit has two phases, which are $Mg(OH)_2$ and $La(OH)_3$.

FTIR analysis confirms the presence of the two phases identified in XRD diffractogram.

This study is a preliminary work to investigate the use feasibility of the obtained samples as potential materials for the hydrogen storage.

Keywords

Mg-La alloys, Electrodeposition, Magnesium hydroxide, Lanthanum hydroxide.

1. INTRODUCTION

Magnesium is the 8th most abundant element in the Earth's crust and the 9th most abundant element in the universe [1].

It is characterized by various properties for instance a low density, a low cost, a very good malleability and ductility and an environmentally friendly nature [2-4]. Magnesium is very chemically active and used in pyrotechnic and incendiary devices.

It is alloyed with other metals such as aluminum, manganese, zircon, zinc, rare-earth metals and thorium. Magnesium and its alloys have the advantageous properties such as low density, high specific strength, good castability, and excellent machinability. They have a broad potential of application and development in marine, automotive, opto-electronic and hydrogen storage materials [5-11]. The addition of rare-earth elements to Mg has attracted considerable attention in the past few years [12-15].

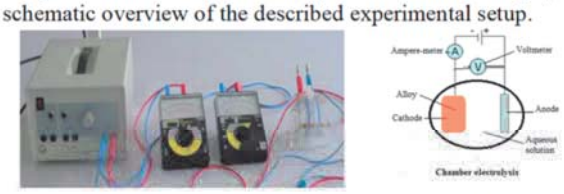
Usually, the elaboration processes of Mg alloys can be classified into three methods: physical, mechanical and chemical. Of these methods, the electrodeposition, which is a much less expensive technique that can be easily implemented on a large scale and can be generally employed over a wide temperature range.

The main objective of this study is to characterize Magnesium-Lanthanum alloys elaborated by means of an electrodeposition technique.

2. EXPERIMENTAL PROCEDURE

Magnesium chloride hexahydrate ($MgCl_2.6H_2O$) and Lanthanum nitrate ($La(NO_3)_3.6H_2O$) from Merck Group were employed as received. The solutions were prepared in deionized water, obtained from a Medica Lab Elga deionizer system. A mass of 16.24 g of $MgCl_2.6H_2O$ and a mass of 4.32 g of $La(NO_3)_3.6H2O$ were dissolved in 34 mL of ultra-pure water at ambient temperature (23 °C). The obtained aqueous solutions with pH=6 were prepared for electrodeposition process.

The electrodeposition process consists in a cylindrical electrolysis chamber. A copper cathode plate and a tungsten thread anode, connected to a conventional potentiometric power source, were used to ensure the deposition of Mg-La alloys. The potential value selected was 3 Volts. The deposition experiment was performed during 8 h at room temperature. Figure 1 shows a photograph and a schematic overview of the described experimental setup.



(a) (b)

Figure 1. Illustrations of the electrodeposition experimental setup. (a) Photograph and (b) schematic overview.

The morphology and chemical composition of the obtained alloy were investigated by a scanning electron microscope (SEM, LEO 1455 VP, acceleration voltage of 20 kV) at a working distance of 15 mm equipped with an X-ray energy dispersive spectrometer (EDS, Inca X- sight, Oxford Instrument). The phase identification of the alloy were determined by means of X-ray diffractometer (XRD, Philips X' PERT) using Cu Kα radiation source (λ=1.54 Å, 40 kV, 30 mA) at room temperature. The scanning rate of 0.02°/s within the 2θ range going from 10° to 90° was used in order to increase counting statistics and to improve the signal/noise ratio. Fourier transform infrared (FTIR) spectroscopy of the alloys samples was carried out with a Nicolet 380 FT-IR spectrometer using the ATR technique. All spectra were collected with the resolution of 4 cm^{-1} in the range of 4000-500 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1 EDS analysis

The result of EDS analysis presented in Table 1 give the elemental composition of the Mg-La alloy. These result illustrate clearly the presence of Oxygen, Magnesium, Lanthanum and very little of Chlorine in the studied sample. The elements O, Mg, La and Cl in the as-deposited powder are because of the electrolyte used in electrodeposition experiment. As we can see from this Table, Mg-La alloys are characterized by high contents of Lanthanum (51.4 w.%) and Oxygen (32.4 w.%).

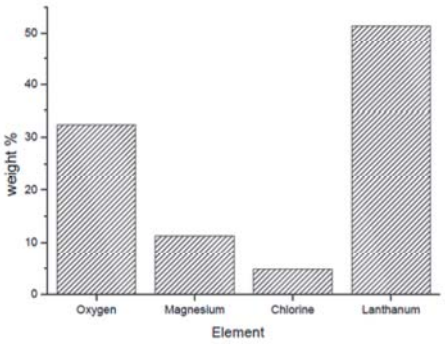


Figure 2. The elemental composition of the obtained alloy in weight percentage.

The morphological characteristics of as-prepared Mg-La alloy are shown in Figure 3. The SEM image exhibits that heterogeneous chemical structures were formed on the surface of the sample. Most of the as-deposited powder shows large aggregates and the different phases seem quite imbricated.

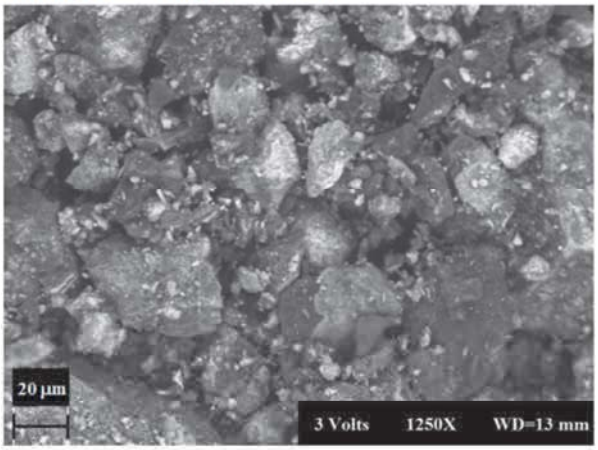


Figure 3. SEM image of the as-deposited powder.

3.2 X-ray diffraction analysis

The different phases of the as-prepared powder are distinguished clearly in Figure 4 through XRD diffractogram. According to the standard data JCPDS, two significant characteristic peaks are detected, which indicate the existence of Magnesium hydroxide ($Mg(OH)_2$) and Lanthanum hydroxide ($La(OH)_3$). As we can see from this Figure, peaks at diffraction angle 2θ of 18.6°, 35.8°, 37.9°, 50.8°, 58.7° and 62.1° are associated to $Mg(OH)_2$ whereas those at 2θ of 15.6°, 27.9°, 39.3°, 46.8°, 48.5° and 54.9° are attributed to $La(OH)_3$.

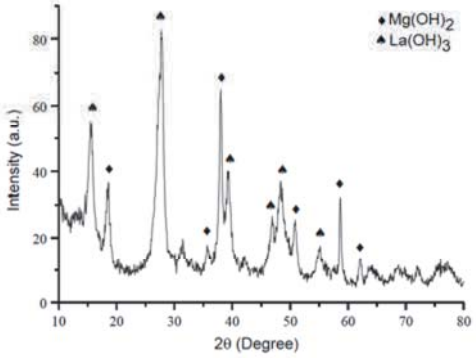


Figure 4. X-ray diffraction pattern of sample.

3.3 FTIR spectroscopy analysis

In addition to the above analyzes, the FTIR spectroscopy study is obvious for the structural confirmation of the as-prepared powder. Figure 5 exhibits the FTIR transition spectrum of the as-deposited Mg-La alloy in the range of 4000-500 cm^{-1} for the electrodepositing voltage of 3 Volts. One can see from this Figure a sharp and strong peak at 3696 cm^{-1} which corresponds to vibrations and stretching of OH bonds in $Mg(OH)_2$ as reported by Jianjun et al. [16] and Gray-Munro and Strong [17]. The band centred at 3606 cm^{-1} is assigned for stretching mode of OH^- in Lanthanum hydroxide [17, 18]. The bands at 3455 cm^{-1} and 1640 cm^{-1} are attributed to the hydroxyl groups in water [17, 19]. The two others distinct bands at about 1434 cm^{-1} and 966 cm^{-1} can be associated to asymmetric stretching vibrations of the carbonate group, which originate from the reaction of the as-deposited powders with CO_2 from air during the FTIR spectroscopy analysis [16, 17, 19].

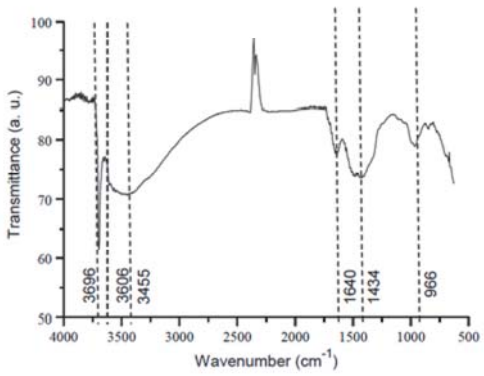


Figure 5. FTIR transition spectrum of the as-prepared powder.

4. CONCLUSION

The preparation of the magnesium-lanthanum powder by electrochemical process at ambient temperature has been successfully treated

As future analyzes, certain parameters would be studied: the concentrations of the electrolyte, the nature of cathode, the current density and electrolysis time; in order to make a comparison and determine the potential material for the hydrogen storage.

5. REFERENCES

[1] A.M. Fekry and R.H. Tammam, Corrosion and impedance studies on magnesium alloy in oxalate solution, *Materials Science and Engineering B*, Vol.176, pp. 792–798, 2011.

[2] B.L Mordike, T Ebert, Magnesium: Properties — applications — potential, *Materials Science and Engineering: A*, Vol. 302, pp. 37-45, 2001.

[3] Kirkland, N T, Magnesium biomaterials: past, present and future, *Corrosion Engineering, Science and Technology*, Vol. 47, pp. 322-328, 2012.

[4] N. Hort, Y. Huang, D. Fechner, M. Störmer, C. Blawert, F. Witte, C. Vogt, H. Drücker, R. Willumeit, K.U. Kainer, F. Feyerabend, Magnesium alloys as implant materials – Principles of property design for Mg–RE alloys, *Acta Biomaterialia*, Vol. 6, pp. 1714-1725, 2010.

[5] V. V. Narulkar, S. Prakash, K. Chandra, Ceramic coated Y1 magnesium alloy surfaces by microarc oxidation process for marine applications, *Bulletin of Materials Science*, Vol. 30, pp. 399-402, 2007.

[6] M. K. Kulekci, Magnesium and its alloys applications in automotive industry, *The International Journal of Advanced Manufacturing Technology*, Vol. 39, pp. 851-865, 2008.

[7] A. Moses Ezhil Raj, C. Ravidhas, R. Ravishankar, A. Rathish Kumar, G. Selvan, M. Jayachandran, C. Sanjeeviraja, Optimized deposition and characterization of nanocrystalline magnesium indium oxide thin films for opto-electronic applications, *Materials Research Bulletin*, Vol. 44, pp. 1051-1057, 2009.

[8] M. Jurczyk, M. Nowak, A. Szajek, A. Jezierski, Hydrogen storage by Mg-based nanocomposites, *International Journal of Hydrogen Energy*, Vol. 37, pp. 3652-3658, 2012.

[9] J. Li , R. Chen, Y. Ma, W. Ke , Effect of Zr modification on solidification behavior and mechanical properties of Mg–Y–RE (WE54) alloy, *Journal of Magnesium and Alloys*, Vol. 1, pp. 346-351, 2013.

[10] A. A. Zaldívar-Cadena, I. Díaz-Peña, J. G. Cabañas-Moreno, Dispersion of niquel on the microstructure in magnesium based alloys for hydrogen storage, *Journal of Magnesium and Alloys*, Vol. 1, pp. 292–296, 2013.

[11] A.A. Luo , Magnesium casting technology for structural applications, *Journal of Magnesium and Alloys*, Vol.1, pp. 2-22, 2013.

[12] J. Kubásek, D. Vojtěch, Structural and corrosion characterization of biodegradable Mg–RE (RE=Gd, Y, Nd) alloys, *Transactions of Nonferrous Metals Society of China*, Vol. 23, pp. 1215-1225, 2013.

[13] T. Liu, Y. Cao, C. Qin, W. Chou, X. Li, Synthesis and hydrogen storage properties of Mg–10.6La–3.5Ni nanoparticles, *Journal of Power Sources*, Vol. 246, pp. 277-282, 2014.

[14] I. Basu and T. Al-Samman, Triggering rare earth texture modification in magnesium alloys by addition of zinc and zirconium, *Acta Materialia*, Vol. 67, pp.116–133 , 2014.

[15] L. Pasquini, E. Callini, M. Brighi, F. Boscherini, A. Montone, T. R. Jensen, C. Maurizio, M. V. Antisari, E. Bonetti, Magnesium nanoparticles with transition metal decoration for hydrogen storage, *Journal of Nanoparticle Research*, Vol. 13, pp. 5727-5737, 2011.

[16] W. Han, Q. Chen, Y. Sun, T. Jiang, M. Zhang, Electrodeposition of Mg-Li-Al-La Alloys on Inert Cathode in Molten LiCl-KCl Eutectic Salt, *Metallurgical and Materials Transactions B*, Vol. 42, pp. 1367-1375, 2011.

[17] B. Xu, M. Zhang, G. Ling, Electrolytic etching of AZ91D Mg alloy in AlCl3–EMIC ionic liquid for the electrodeposition of adhesive Al coating, Surface and Coatings Technology, In Press, 2013.

[18] L. Jianjun, Y. Xiaoyan, C. Jia, L. Xiaowei, F. Chengxing, C. Youfa, The gemmological properties and infrared spectra of brucite, an imitation of nephrite and Shoushan stone, *The Journal of Gemmology*, Vol.32, pp. 67-73, 2010.

[19] J.E. Gray-Munro, M. Strong, A study on the interfacial chemistry of magnesium hydroxide surfaces in aqueous phosphate solutions: Influence of Ca^{2+}, Cl^- and protein, *Journal of Colloid and Interface Science*, Vol. 393, pp. 421-428, 2013.