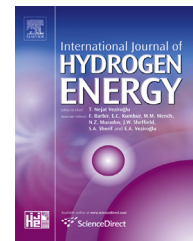




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The electrochemical hydrogen storage properties of $\text{Ti}_{0.72}\text{Zr}_{0.28}\text{Mn}_{1.6}\text{V}_{0.4}$ alloy synthesized by vacuum plasma spraying and vacuum copper boat induction melting: A comparative study

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ARTICLE INFO

Article history:

Received 18 May 2015

Received in revised form

27 August 2015

Accepted 1 September 2015

Available online 23 October 2015

Keywords:

Hydrogen storage alloys

Vacuum plasma spraying

Vacuum induction melting

Electrochemical characterization

ABSTRACT

In this study, two different rapid solidification techniques; vacuum plasma spraying and vacuum copper boat induction melting, were performed to synthesize $\text{Ti}_{0.72}\text{Zr}_{0.28}\text{Mn}_{1.6}\text{V}_{0.4}$ hydrogen storage alloy. The structure and electrochemical properties of the produced samples were comparatively investigated. The chemical and phase compositions of the samples were characterized by XRD and SEM analysis. The electrochemical hydrogen storage characteristics including the cyclic stability, the maximum discharge capacity, the high rate dischargeability, and the electrochemical impedance spectra (EIS) were measured. Results indicated that both samples consisted of a hexagonal C14 Laves phase matrix and a body centered cubic (BCC) structure of V-based solid solution phase. The electrochemical results showed that in the alloy prepared by induction melting, the maximum discharge capacity was higher, while the cyclic stability of the vacuum plasma sprayed sample was much better. These results were attributed to the quantity and distribution of the presence phases and the anti-corrosion performance of the alloys. Moreover, the results from the electrochemical kinetic experiments showed that the vacuum plasma sprayed sample had superior kinetic parameters, including the hydrogen diffusion rate and charge-transfer resistance, than that for the vacuum induction melted sample. Thus, vacuum plasma spraying could be an efficient production method for preparation of hydrogen storage alloys with improved properties which could be used for Ni–MH secondary batteries as negative electrodes.

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<http://dx.doi.org/10.1016/j.ijhydene.2015.09.081>

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Introduction

At present, environmental and economical problems related to fossil fuels consumption, CO₂ emission and global warming have motivated the researches for developing new, environment friendly, comparable to fossil fuels, renewable alternative energy sources. In this way, hydrogen as an ideal energy carrier has a high potential to be a renewable substitute for transportation and domestic applications because of substantial chemical energy per mass (142 MJ/kg), environment safety and availability. However, the lack of a safe, efficient, and inexpensive hydrogen storage media is the main challenge [1–4]. Among the various hydrogen storage methods, solid state storage using metals and alloys in shape of metal/alloy hydrides has attracted more attention. These alloys are utilized either in the hydrogen storage tanks for fuel cell vehicles or throughout electrochemical hydrogen storage method in nickel–metal hydride (Ni–MH) secondary batteries as the negative electrode materials [5–7]. Currently, the electrochemical hydrogen storage method is more applicable in many applications in which hydrogen as atomic form directly diffuses into the metallic phase through electrochemical decomposition of water molecules at ambient temperature and atmospheric pressure [8]. Several types of metal hydride alloys have been studied as negative electrode material including rare earth-based AB₅ type alloys [9,10], Ti, Zr-based AB₂ alloys [11–15], Mg-based alloys [16–18], V-based solid solution [19–22], rare earth-based AB₃ type alloys [23,24] and Ni–Ti series [25,26]. Nowadays, AB₅ type alloys such as LaNi₅, which are operated at moderate temperature and pressure, are commercially used in the Ni/MH batteries. However, their hydrogen storage capacity does not exceed 1.4 wt % [27]. Other hydrogen storage alloys such alkali and light based metal hydrides have more hydrogen storage capacity. In these groups, Mg-based alloys show hydrogen storage capacity in the range of 2–7 wt % but at the high unfavorable temperatures (higher than 200 °C) and with slow desorption kinetics. Alkali metals group such as Li-based alloys or sodium alanates also have more hydrogen storage capacity (2.5–10 wt%), however these alloys are not considered as rechargeable hydrogen storage alloys due to their weak reversibility, high equilibrium pressure (>20 bar) at room temperature, high decomposition temperature (~250–350 °C) and poor kinetics. Thus, the hydrogen desorption can last up to nearly 4000 min and hence these alloys still could not be commercially applicable [28,29]. Among all the above mentioned alloys, AB₂ alloys are good candidates to replace mischmetal based AB₅ alloys used as metal hydride electrode at Ni/MH batteries because of their higher storage capacity, faster kinetics, longer life and relatively low cost [30]. The potential AB₂ type alloys are prepared by Ti and Zr on the A site and various combinations of transition metals, especially V, Cr, Fe and Mn on the B site. The hydrogen storage capacity of these alloys can reach to more than 2 wt% at ambient temperature and atmospheric pressure [31]. Ti-based AB₂ alloys such as TiMn₂ have received further attention due to their higher discharge electrochemical capacity, easier activation, cycling stability and flexibility in composition design [7,32–34]. However, fast decrease in their discharge capacity within several

charge–discharge cycles and their low high-rate discharge ability are the main obstacles for high volume commercial production [35–37]. In the recent years, the hydrogen storage characteristics of the alloys were improved by several methods such as: (1) adding alloying elements or components with catalytic effects [38], (2) modifying the surface properties of the hydrides [39], (3) employing new preparation methods including, smelting methods, mechanical alloying, hydriding combustion synthesis, sputter method, physical vapor deposition (PVD), melt-spinning, severe plastic deformation (ECAP, cold rolling, ARB), gas atomization and solid phase sintering [40–48]. Nowadays, development of novel mass production methods is one of the important research challenges.

Electrochemical and gaseous properties of hydrogen storage alloys are effectively related to the structure of the existent phases, phase abundance, and microstructure parameters correlated to the alloy production process such as particles morphology, chemical homogeneity, and grain size of the material [47].

Up to now, in many investigations, alloy smelting has been the most common method for preparing hydrogen storage alloys [41]. For an ingot prepared by prevalent methods such as arc and induction melting, the segregation occurs during the alloy ingot solidification. The newer methods such as melt spinning and gas atomization with their high cooling rates and also mechanical alloying may produce ingots and powders with a better chemical homogeneity and smaller grain size than those synthesized by conventional casting methods, that leads to improve hydrogen storage characteristics, particularly the hydrogen absorption and desorption kinetics and cyclability [36,47,49–51].

The relatively novel vacuum plasma spray process; a rapid solidification and commonly used deposition technique in the industry, can be a promising method for mass production of the hydrogen storage alloys with suitable properties. In a research by Cakmak et al. [52] the plasma synthesis was applied for preparing Mg–Ti alloy and the results showed this method was very beneficial for production novel hydrogen storage alloys.

In this research, a new composition of TiMn₂-based alloys (Ti_{0.72}Zr_{0.28}Mn_{1.6}V_{0.4}) was considered based on the results obtained from the previous investigations [44,53–56]. The alloy was prepared by two production methods: vacuum plasma spraying (VPS) and copper boat induction melting (CBIM). After production, the structure and electrochemical properties of the samples were investigated.

Materials and methods

The alloy used in this study was prepared by two methods: vacuum plasma spraying (VPS) and vacuum copper boat induction melting (CBIM). The purity of constituent elements was above 99.9%. Before VPS deposition, the alloy elements were mixed by ball milling for 4 h in a Retch PM100 planetary ball mill under the ball to powder weight ratio of 15:1 with five 30 g steel balls and with rotational speed of 200 rpm under argon atmosphere. The powder average particle size after milling was 45 μm. The VPS was carried out at the Centre for Advanced Coating Technologies at the University of Toronto. The vacuum plasma spray process parameters are given in

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