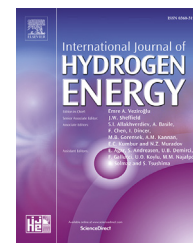


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Study the effect of SrFe₁₂O₁₉ on MgH₂/LiAlH₄ composite for solid-state hydrogen storage

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ARTICLE INFO

Article history:

Received 8 August 2017

Received in revised form

2 October 2017

Accepted 12 October 2017

Available online 7 November 2017

Keywords:

Hydrogen storage

Destabilized system

Magnesium hydride

Alanate

Catalyst

ABSTRACT

The MgH₂/LiAlH₄ destabilised system was studied experimentally using the method of ball milling. The desorption/absorption performances and reaction mechanism of the 4MgH₂ + LiAlH₄ composite system with SrFe₁₂O₁₉ additive have been investigated for the first time. Analysis of the temperature-programme-desorption showed that the 5 wt% SrFe₁₂O₁₉-doped 4MgH₂ + LiAlH₄ composite started to release hydrogen at 80 °C and 260 °C for the first two desorption stages, which were reduced by 40 °C and 10 °C as compared to the undoped composite. The sorption kinetics of 5 wt% SrFe₁₂O₁₉-doped 4MgH₂ + LiAlH₄ were also improved as compared to the undoped 4MgH₂ + LiAlH₄. Activation energy calculation based on the Kissinger plot displayed that the apparent activation energy for the decomposition of MgH₂-relevant had been reduced from 121 kJ/mol for the undoped composite to 104 kJ/mol for the composite with SrFe₁₂O₁₉. The X-ray diffraction results suggested that the new species of Al₂Sr and Li₂Fe₃O₄ that were formed in situ during the heating process were believed to play a catalytic role, thus responsible for the enhancement of the hydrogen storage properties of 4MgH₂ + LiAlH₄ composite system with SrFe₁₂O₁₉.

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Introduction

As we move forward to using clean energy sources, hydrogen, which provides energy without pollution (produces a clean form of energy when burnt in air), is an ideal candidate to fuel our future energy needs. In addition, when compared with gasoline and methane in terms of the safety aspects of fuels, hydrogen is the safest fuel [1]. Hydrogen can thus be considered as one of the versatile and environmentally compatible energy carrier, mainly in fuel energy application. On the other hand, there are three major steps of the hydrogen-energy process chain, namely; hydrogen production, hydrogen storage and repowering, as have been studied in the previous

studies [2–4]. Nevertheless, the storage of hydrogen is a major bottleneck in its use. On the other hand, according to the U.S. Department of Energy (DOE), 6.5 wt% of the hydrogen-storage capacity at the dehydrogenation temperature between 60 and 120 °C together with high life cycle is targeted for commercialisation [5]. Hence, the issue related to hydrogen storage has to be overcome by finding the best method to store hydrogen to meet the goals of U.S. DOE for transport application.

Conventionally, hydrogen is stored as compressed hydrogen gas (in high pressure cylinders), cryogenic liquid (in cryogenic containers) or solid state (chemical compounds that reversibly release hydrogen upon heating). However, a lot of space and a huge storage system are required for hydrogen storage in gaseous and cryogenic liquid states [6]. Because of

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the foregoing, solid-state hydrogen storage that possesses several advantages, such as large gravimetric storage density, cost effectiveness, preferable safety and more space for hydrogen storage [7], has received considerable interest as a potential hydrogen-storage medium.

Chemically bound hydrogen (chemisorption) and physically bound hydrogen (physisorption) are two types of solid-state hydrogen-storage materials. Among them, metal hydride based on chemisorptions has been the focus of intensive research. Magnesium hydride (MgH_2) is a classic metal hydride and is considered as one of the most promising candidates that can be used for transportation applications owing to its large gravimetric density (7.6 wt%), low cost and superior reversibility. Nevertheless, the high temperature of hydrogen discharge (300 °C at 1 bar pressure) and sluggish sorption kinetics of MgH_2 become the major hurdles in the widespread use for mobile storage applications [5,8]. Thus, various methods have been employed to address the limitations and enhance the hydrogenation properties of MgH_2 ; namely, altering the size of the particles by ball milling [9,10], addition of a catalyst, which can facilitate the hydrogen sorption kinetics [11–15] and alloying with other metal/complex hydrides (destabilisation concept) [16–28]. The destabilisation concept, also known as reactive hydride composites method, can be achieved by altering the thermodynamics and reaction of the hydrogen sorption kinetics, which results in the formation of intermediate phases that may help the process of thermodynamic and kinetic properties of hydrogen release and uptake [29–32].

Therefore, researchers have endeavoured to destabilise MgH_2 with the most studied systems under complex hydrides (lithium aluminium hydride powder, LiAlH_4) for ameliorating the obstacles in the practical use of MgH_2 and enhancing the hydrogenation properties of MgH_2 and LiAlH_4 [31,33]. LiAlH_4 of the alanates family has received considerable interest as one of the promising potential materials to store hydrogen because of its superior intrinsic theoretical gravimetric storage capacity of 10.6 wt% H_2 [34] and high reversible hydrogenation ability [35].

Among all the promising hydride composites, $\text{MgH}_2 + \text{LiAlH}_4$ destabilised system has been the focus of intensive research and seems to have the potential to rise to the expectations of DOE for mobile applications. Recently, numerous studies have reported the positive feedbacks of the reaction mechanism and thermodynamics of the $\text{MgH}_2 + \text{LiAlH}_4$ catalyst system [36,37]. However, persistent endeavours are needed to investigate another potential catalyst to promote the hydrogen de/rehydrogenation performance of the $\text{MgH}_2 + \text{LiAlH}_4$ composite system as a suitable hydrogen-storage medium. To the best of our knowledge, the role of a ferrite-based complex metal oxide (ternary oxide) as one of the efficient catalysts of the Li–Mg–Al–H system has not yet been extensively explored. A study conducted by Wan et al. [37] reported that the reactions between MnFe_2O_4 and the $\text{MgH}_2 + \text{LiAlH}_4$ destabilised system could enhance the hydrogen sorption performance of the composite system. Motivated by the above findings, more studies are needed to find another efficient catalyst from the ferrite group that have been revealed to possess the best catalytic efficiency.

More recently, the authors studied the effects of another type of ferrite, strontium ferrite ($\text{SrFe}_{12}\text{O}_{19}$), on the hydrogen sorption behaviour of MgH_2 [38]. We found that the new phases Fe, MgFe_2O_4 and SrO that were formed during the heating process might act as the active species, which further improved the onset decomposition temperature as well as the re/dehydrogenation kinetics of MgH_2 . Hence, it is reasonable to hypothesise that Sr ferrite could display an extremely great potential as a catalyst for the $\text{MgH}_2 + \text{LiAlH}_4$ destabilised system to advance the onset decomposition temperature and de/rehydrogenation properties. The present study utilised $\text{SrFe}_{12}\text{O}_{19}$ as a catalyst to investigate its effect on the hydrogenation performance of the $\text{MgH}_2 + \text{LiAlH}_4$ destabilised system. To date, there has been no report of $\text{SrFe}_{12}\text{O}_{19}$ doping $\text{MgH}_2 + \text{LiAlH}_4$ for solid-state hydrogen storage. The possible catalytic mechanism from the results obtained is discussed herein.

Experimental details

Pure MgH_2 (hydrogen-storage grade, 98% purity), LiAlH_4 (95% purity) and $\text{SrFe}_{12}\text{O}_{19}$ (<100 nm particle size (BET), 99.8% trace metals basis) were purchased from Sigma-Aldrich. All the raw materials were used in as-received state with no additional purification. All handling (including weighing and loading) was performed in a high-purity argon-filled MBraun Unilab glove box (H_2O and O_2 : <0.1 ppm) to minimise oxidation and moisture. The ball milling process of the MgH_2 and LiAlH_4 powders in a mole ratio of 4:1 was conducted in a planetary ball mill (NQM-0.4) for 60 min at a rotating rate of 400 rpm using a stainless steel vial together with four hardened stainless steel balls. After every 15 min of ball milling, the steel vial was rested for 2 min to cool it down and then milled for another 15 min for 2 cycles. For simplicity, this composite will be referred to as $4\text{MgH}_2 + \text{LiAlH}_4$. The sample of $4\text{MgH}_2 + \text{LiAlH}_4$ added with 5 wt% $\text{SrFe}_{12}\text{O}_{19}$, as well as pure MgH_2 and LiAlH_4 , were prepared under the same conditions for comparison.

Temperature-programmed desorption (TPD) and absorption and desorption kinetics experiments were conducted in Sieverts-type pressure-composition temperature (PCT) apparatus (Advanced Material Corporation). The PCT instrument was connected to a computer and was controlled by GrcLV software that performed fully automatic operations. About 100 mg of the sample was loaded into a sample vessel, which conducted in the glove box. For desorption, all the samples were heated from 25 °C to 450 °C in a vacuum chamber at a heating rate of 5 °C/min under controlled vacuum of 0.1 atm. The rehydrogenation and dehydrogenation kinetics experiments were conducted at a temperature of 320 °C with the initial hydrogen pressure of 33.0 and 1.0 atm, respectively. The capacity of hydrogen was expressed in weight percentage (wt%).

The reaction mechanism of the samples after ball milling, dehydrogenation and rehydrogenation were studied using X-ray diffraction (XRD; Rigaku MiniFlex II diffractometer with $\text{Cu K}\alpha$ radiation). θ – 2θ scans were varied from 20° to 80° with a scanning velocity of 2.00°/min. The surface morphology of all the samples studied were characterised using scanning

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