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Hydrogen generation by aluminum hydrolysis using the Fe₂Al₅ intermetallic compound

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ABSTRACT

Intermetallic phases with high aluminum content release hydrogen naturally; this is due to their susceptibility to hydrogen environmental embrittlement. Thus, in this work we take advantage of this phenomenon to assess hydrogen generation using the Fe₂Al₅ intermetallic that is mechanically activated. Furthermore, various aqueous solutions were evaluated by adjusting the pH with NaOH and CaO additions to increase both the rate and the amount of hydrogen generation. The Fe₂Al₅ intermetallic compound is useful material for the reaction with water and releases hydrogen. The results indicated that as the pH of the solution increases the amount and rate of hydrogen production increases. Reaction efficiency tends to be higher at pH = 13 with NaOH additive (near to 100%) being clearly better than CaO additive (35%). Small reaction capabilities with CaO additions are probably due to the CaO total consumption at the initial stages of the reaction, as the bayerite phase (reaction byproducts) is formed. At pH = 14, the NaOH addition reacts with iron, in addition to the reaction with aluminum particles, to achieve more than 100% reaction efficiency according to the presence of an iron oxyhydroxide phase identified by X-Ray diffraction.

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Introduction

Historically, hydrogen technology is considered a source of clean and renewable energy [1,2]. However, the safe storage of hydrogen presents significant research challenges. Hydrogen has the best fuel capacity, but we must live with the risk of its storage in pressurized tanks, especially for applications in mobile devices [3]. A potential alternative to solving the storage problem is to produce hydrogen at room temperature on demand in a cell filled with metals that react with water. We find that pure aluminum and its alloys have been identified as the most suitable materials to generate hydrogen from water

[4–7]. Aluminum and its alloys have low costs and low density. One gram of aluminum can release 1.24 L of hydrogen, according to equation (1) [5].



However, pure aluminum does not react with water by itself due to the existence of an aluminum oxide film on its surface. So a chemical activation must be performed. Three main aluminum activation methods have been previously employed to evaluate hydrogen generation. First, aluminum alloyed with certain metals including Ga, In, and Hg [8–10]. Second, the uses of additives like NaCl, Al₂O₃, and C [11–14]. The third method is the use of alkaline solutions that modify

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the pH, such as CaO, NaOH and KOH [15–17]. Another successful method for aluminum chemical activation is mechanical milling, which promotes the reaction because the crystal size reduction exposes a new surface area of the material [6,18,19]. Also, the ball-milling introduces point defects that improve the diffusion and remove the oxide layer that impedes water molecules from diffusion inside material [12,20]. On the other side, it is well-known that the intermetallic compounds are susceptible to hydrogen environment embrittlement [21,22]. That is, intermetallic materials at room temperature react spontaneously with moisture in the air to release hydrogen. The reaction produces monatomic hydrogen, which reduces the cohesive forces, leading to cleavage fracture of the intermetallic compound and exposing the new surface area to the phenomenon [23,24]. This chemical behavior is the primary reason why intermetallic materials have poor mechanical properties, affecting its structural applications. To date, little research has been developed for evaluating aluminum intermetallic alloys as a possible material for hydrogen generation. Thus, in the present study, hydrogen generation results from the reaction between aluminum from the Fe_2Al_5 intermetallic compound and water. The intermetallic alloy was previously mechanically activated by ball-milling. Moreover, the variation of the pH of the water is also evaluated by the additions of NaOH and CaO.

Experimental

Materials

The starting materials were: Al powder (99% purity), Fe powder (99% purity), NaOH (98% purity), and CaO (96% purity). The metals and the chemical substances were from Alpha Aesar Sinopharm Chemical Reagent and Sigma–Aldrich. Deionized water was used to prepare all the aqueous solutions.

Alloy intermetallic preparation

We prepare Fe_2Al_5 intermetallic alloy by melting Al and Fe elements in an induction furnace. The as-cast ingot was crushed into particles of ~5 mm and milled for 1 h, 5 h, and 10 h in an SPEX 8000M mixer mill using hardened steel vials and balls. The milling speed was 1800 rpm. The weight of the intermetallic powders charged in the container was 3 g to obtain a ball-to-powder weight ratio of 7:1. After the milling process, particles were stored in glass containers in the air for different time periods. The effect on the H_2 generation of both milling and storage time was observed and discussed in the present investigation.

Hydrogen measurements

The experimental procedure to determine the hydrogen gas released was carried out using the displacement method. Fe_2Al_5 intermetallic powders were placed in a 200 ml acrylic-cylindrical reactor. The mass of Al from the intermetallic compound was 0.33 g, which is equivalent to produce 250 ml H_2 under standard conditions (298 K and 1 atm.), assuming 100 percent efficiency. Different aqueous solutions containing

CaO and NaOH were prepared by pH adjusting at 12–14. Then, 20 ml of the reaction solution was added to the activated powders. The H_2 gas under standard was measured as the water displacement in an inverted burette. The time to the first H_2 generated was measured by observing when the first bubble formed in the burette. The induction time, the rate of generation, and the total amount of hydrogen released were measured for each experiment.

Microstructural characterization

The microstructural characterization of the as-cast, milled and treated samples was performed using different structural characterization techniques, including X-ray diffraction (XRD) with $\text{CuK}\alpha$ radiation (Siemens D5000 X-ray diffractometer), scanning electron microscope (Jeol, JSM 2400), and transmission electron microscopy (Philips Tecnai F20 at 200 kV).

Results and discussion

Fig. 1 shows the hydrogen production curves of the Fe_2Al_5 intermetallic powder corresponding to the solution of pH = 12 with 1 h, 5 h, and 10 h of milling times. After the initiation of the reaction, the hydrogen-generation rate increased exponentially with the milling time. However, a small variation in the hydrogen generation rate and induction time with increasing milling time was obtained. At 10 h of milling, the longer induction period of approximately 15 min, which may be due to prolonged milling times, lead to oxidation of the material causing a reduction in the amount of hydrogen released. Fan et al. has reported similar trends [25], where the reactivity of the alloy decreases at higher milling times, up to 5 h. Based on these results, 1 h of milling was selected to carry out the subsequent experiments.

Fig. 2 displays images of scanning electron microscopy showing the morphology and particle size of the by-products with increasing milling time of Fe_2Al_5 (1 h, 5 h, and 10 h). For

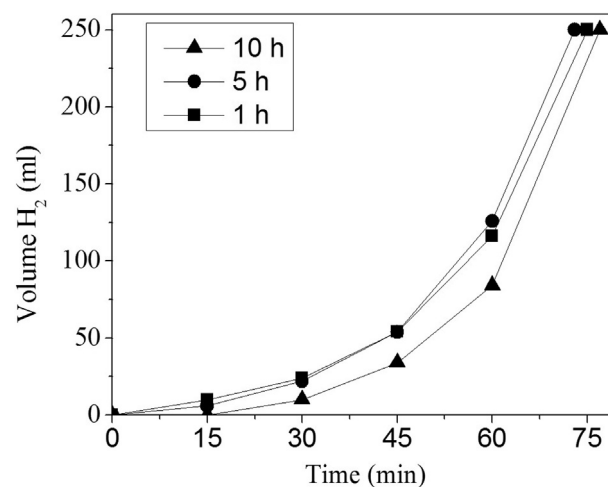


Fig. 1 – Hydrogen generation curves, varying the milling time of Fe_2Al_5 intermetallic using NaOH solution of pH = 12.

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