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A novel material of nanoporous magnesium for hydrogen generation with salt water

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HIGHLIGHTS

- Nanoporous Mg is successfully prepared by a physical vapor deposition method.
- Three-dimensional structure of the nanoporous Mg is successfully characterized.
- Nanoporous Mg can generate hydrogen with salt water directly.
- Hydrogen generation property of nanoporous Mg is better than present materials.
- The reason of the excellent hydrogen generation property has been revealed.

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ABSTRACT

In this paper, two kinds of nanoporous Mg are prepared by a physical vapor deposition method, and their hydrogen generation properties are investigated based on the hydrolysis reaction with salt water. The results indicate that cell-like nanoporous Mg has higher hydrogen generation amount of 800 ml g^{-1} and hydrogen generation rate of $48.1 \text{ ml g}^{-1} \text{ min}^{-1}$ than block-like nanoporous Mg, while block-like nanoporous Mg with the substrate shows better hydrogen generation property of 933 ml g^{-1} and $78.4 \text{ ml g}^{-1} \text{ min}^{-1}$. The effects of concentration of salt water and temperature on their hydrogen generation property have been investigated. In order to explain their excellent hydrogen generation properties, electrode potential and specific surface area of different Mg materials are examined. The results indicate that cell-like nanoporous Mg has a more negative potential (-2.410 V (vs.RHE)) than block-like nanoporous Mg (-2.279 V (vs.RHE)), and a higher specific surface area of $26.7 \text{ m}^2 \text{ g}^{-1}$ than block-like nanoporous Mg of $0.9 \text{ m}^2 \text{ g}^{-1}$, both of which are more negative and higher than that of Mg plate and Mg alloy plate. Thus, it can be concluded that more negative potential and higher specific surface area of nanoporous Mg are the reasons for their better hydrogen generation property.

1. Introduction

The energy crisis and environmental pollution caused by fossil fuel consumption make it urgent to develop new energies. Hydrogen energy is considered to be one of the most attractive new energy due to its high energy density, regeneration and environmentally friendly character [1–3] and becomes the focus of nowadays study. As the carrier of hydrogen energy, hydrogen gas is the main form of performance, and it can be used either to produce the heat energy through combustion reaction, such as rocket launching system [4,5], or to produce the electrical energy through electrochemical reaction, such as fuel cell power system [6–11]. About the hydrogen generation, many methods, such as decomposition of fossil fuels [12–14], water decomposition [15–17]

and biohydrogen production [18,19], are currently employed. However, these methods are complicated and costly. Furthermore, how to storage and transport hydrogen is another key issue, which connected with the large-scale storage facility and discontinuous supply of hydrogen.

A new method of onsite hydrogen generation has received more and more attention recently, in which hydrogen can be immediately produced by a rapidly hydrolysis reaction [20–24]. Although the output of this method is limited, it is simple, cheap and easy to operate, and is efficient for the hydrogen supply on some special occasions, such as in the wilderness. There are several kinds of materials that can hydrolyze with water, such as aluminum based materials, boron-based hydrides and magnesium based materials [25–28]. The aluminum based

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materials can supply a high amount of hydrogen, but the alumina film that formed on the Al surface hinders the hydrolysis reaction. By the addition of composites, such as LiH, NaBH₄, alkaline salt [29–31], and by addition of alloy elements, such as Fe, Co, Ni [32], this problem can be solved to some extent, however, these additions will enlarge the preparation process and increase the cost. The boron-based hydrides, such as sodium borohydride, will hydrolyze with water when there are suitable catalysts. But these catalysts, such as Pt, Pd and Ru [33–35], are precious and will increase the cost. Magnesium based materials can also hydrolyze with water, but the byproduct of magnesium hydroxide will deposit on the surface of Mg samples, which slows down the reaction. To remove the deposited layer, acid solution was employed [36], but it is environmental unfriendly.

In this study, we got two kinds of novel nanoporous magnesium materials that can hydrolyze with salt water efficiently and continually. These nanoporous Mg materials, block-like nanoporous (BNP) Mg and cell-like nanoporous (CNP) Mg were prepared by a physical vapor deposition method (PVD). And their hydrogen generation properties with salt water were investigated. Furthermore, in order to explain their excellent properties of hydrolysis reaction, polarization curves and specific surface area of these two nanoporous Mg materials were examined and compared with the traditional magnesium based materials.

2. Experimental

The preparation of the block-like nanoporous (BNP) Mg and cell-like nanoporous (CNP) Mg is schematically shown in Fig. 1. The magnesium powders with a purity of 99% and a granularity of ~75 μm were used. These magnesium powders were placed at the bottom of the stainless steel container. A 304 stainless steel wire cloth of 1500 mesh was placed above the Mg powder as the substrate for the deposition of magnesium vapor. For the preparation of BNP Mg, Mg powders were vaporized at 500 °C in a vacuum of 10⁻¹Pa, and the substrate was placed 11 cm high above the Mg powders. The temperature of the substrate was about 200 °C. For the preparation of CNP Mg, Mg powders were vaporized at 550 °C in an ethanol atmosphere of 3Pa, and the substrate was placed 12 cm high above the Mg powders. The time for vaporization and deposition sustained 2 h for both of them.

The scanning electron microscope (Zeiss Auriga) and X-ray diffractometer (TTR III) were used to characterize the morphologies and structures of these nanoporous Mg. The synchrotron radiation (Beijing Synchrotron Radiation Facility, 4W1A-X-ray imaging station, with the spatial resolution of 100 nm) was used to characterize the three-

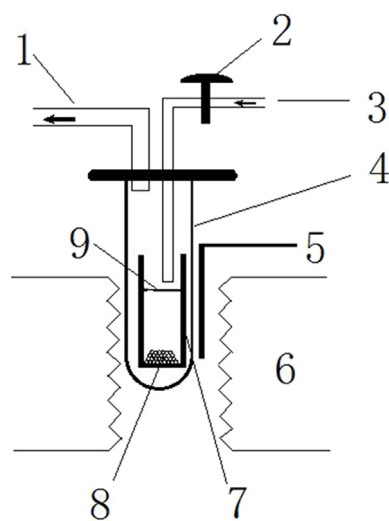


Fig. 1. Scheme for the preparation of nanoporous Mg: 1-pump, 2-gas valve, 3-ethanol steam, 4-quartz tube, 5-thermocouple, 6-furnace, 7-stainless steel container, 8-Mg powders, 9-substrate.

dimensional structures of these nanoporous Mg. By using Amira and Avizo software, the three-dimensional space structures of these nanoporous Mg were reconstructed and the corresponding porosities were calculated.

The specific surface area of these nanoporous Mg was determined by the nitrogen adsorption/desorption tests, in which specific surface area analyzer of Quantachrome and BET method were employed.

Hydrogen generation abilities of these nanoporous Mg materials were determined by salt water immersion test. The nanoporous Mg materials (CNP Mg and BNP Mg), together with Mg plate and Mg alloy (AZ61), were used for these tests under the 10 mass % NaCl solution at 25 °C. Meanwhile, the effects of substrate, solution concentration and temperature on the hydrogen generation abilities of BNP Mg were evaluated. The substrate was stainless steel wire cloth. The BNP Mg with and without substrate were examined under the condition of 10 mass % NaCl solution at 25 °C. The BNP Mg with substrate were also examined under the NaCl solution concentrations of 0%, 3.5%, 5%, 7% and 10 mass % s at 25 °C, and under the temperatures of 0 °C, 25 °C, 35 °C, 50 °C in 5mass% NaCl solution. Based on the hydrogen generation data at different temperatures the activation energy was calculated using Arrhenius equation. By collecting the hydrogen gas (displacement of water) and recording the hydrogen volume at 10 s intervals, the hydrogen generation amount and rate can be obtained. The samples of 0.5 g and the 20 ml solutions were used during this test.

Tafel curves of BNP Mg, CNP Mg, Mg plate and AZ61 plate were obtained using a CHI 660E electrochemical workstation at room temperature, with an electrolyte of salt water (10 mass % NaCl). During the tests, Mg samples with 1 cm*1 cm square were used as the working electrode, Pt as the counter electrode, and Hg/HgO as the reference electrode. The measurement volt is scanned from -3.2 V to -1.2 V at 0.01 mV s⁻¹. The electrode potential E and corrosion current I_{corr} can be calculated from the Tafel curves [37].

3. Results and analysis

3.1. Characterization

The different morphologies of nanoporous Mg, BNP Mg and CNP Mg, are shown in Fig. 2. It can be seen from Fig. 2 (a1), (a2) that the BNP Mg consists of block-like grains. The size of these grains is about 40 μm in diameter. On the grain surface, there exist lots of nanopores. From Fig. 2 (b1), (b2) it can be seen that the CNP Mg has a cell-like morphology and within the ligament, there exists lots of loose nanoporous. By the XRD test, see Fig. 3, it is confirmed that both of BNP Mg and CNP Mg are just the Mg metal, not magnesium oxides.

Fig. 4 shows the three-dimensional structures of the BNP Mg and the CNP Mg determined by synchrotron radiation imaging. The size and distribution of the nanopores in the three-dimensional structure are also demonstrated. By the calculation using Amira and Avizo software, the diameter of the nanopores in BNP Mg is about 200 nm, and the porosity of BNP Mg is about 18.6%, while the diameter of the micropores in CNP Mg is about 1.5 μm, and the porosity of CNP Mg is about 72.0%. Due to the diameter of the micropores in CNP Mg is calculated based on the two kinds of hierarchical pores, which are micro pores and nano pores, the diameter of the micropores in CNP Mg (1.5 μm) is only for reference. However, the porosity of CNP Mg is not influenced by the hierarchical pores, thus it can be concluded that the CNP Mg has a higher porosity than BNP Mg.

The results of nitrogen adsorption/desorption tests for the BNP Mg and CNP Mg can be used to calculate their specific surface area. The specific surface area for the BNP Mg is 0.9 m² g⁻¹ and for the CNP Mg is 26.7 m² g⁻¹, which is higher than the BNP Mg.

3.2. Hydrogen generation property

The hydrogen generation curves of BNP Mg, CNP Mg, Mg plate and

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