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Supplementary Information

Atmospheric self-evolution and hydrogen storage of $Mg_{80}Ni_{20}H_x$ induced by wet ball milling

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Experimental methods

Raw Materials

The following chemicals were used as received commercially without any further purification: Mg powder (≥ 99 %, WeiHao Magnesium Powder, < 44 μm), Ni powder (≥ 99.9 %, Jiangyou Hebao Nanomaterial Co., < 2 μm), acetone (≥ 99.5 %, Shanghai Lingfeng Chemical Reagent Co.).

Sample preparation

Mg-Ni based hydride composites were manufactured by hydriding combustion synthesis (HCS) method. Briefly, the Mg powder and Ni powder were mixed at 80: 20 molar ratio, and then the mixed powder was blended with acetone and dispersed by ultrasound for 1 h. After that, the mixture was dried for 10 h. The as-obtained powder mixture was put into a combustion synthesis furnace, and heated at 535 °C for 1 h under 2 MPa hydrogen atmosphere, then cooled down to 340 °C and held for 4 h under 2 MPa hydrogen atmosphere to obtain $Mg_{80}Ni_{20}H_x$.

Then, as-synthesized $Mg_{80}Ni_{20}H_x$ was partially dry milled and partially wet milled, respectively. In particular, the dry ball milling process was set with a ball to material ratio of 30:1 and a milling speed of 400 rpm, while the wet milling process was set with the same ball to material ratio and milling speed, adding 2 mL of acetone as solvent during milling. After wet ball milling, the samples were oven dried at 60°C. The as-synthesized samples were then exposed to ambient atmosphere for varying periods of time.

Characterizations

X-ray diffraction (XRD, ARL X'TRA diffractometer, Cu-Kα radiation) was used to analyze the phase structure of the samples. The micromorphology was observed by field-emission scanning electron microscopy (FESEM) and high-resolution transmission electron microscopy (HRTEM). The programmed temperature-raising desorption (TPD) and hydrogen absorption/desorption kinetic experiments were measured on a sieverts-type device (GRC, Advanced Materials Co.). For TPD experiments, the samples were heated from room temperature to 360 °C at a heating rate of 10 °C/min. For the isothermal hydrogen absorption/desorption kinetics experiments, the initial hydrogen pressures for hydrogen absorption/desorption were 3 MPa and 0.005 MPa, respectively. The samples were analyzed by differential scanning calorimetry (DSC, TA Q2000) to investigate the hydrogen desorption behavior. About 5 mg of the sample was loaded into a ceramic crucible and then heated to 500 °C under argon atmosphere at a flow rate of 50 ml/min.