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Effect of initial powder type on the hydrogen storage properties of high-pressure torsion consolidated Mg

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ABSTRACT

While severe plastic deformation (SPD) on bulk samples has been widely applied for modifying the H-sorption properties, there has been little attention towards the use of SPD on powder materials. In this context, the aim of the present work was to compare the H-storage properties of high-pressure torsion (HPT) consolidated products obtained from two distinct Mg powder precursors: atomized micro-sized and condensed ultrafine powder particles. The results showed that the nature of the initial powder precursor had a pronounced effect on the H-sorption behavior. The HPT product obtained from the condensed ultrafine powder showed faster absorption kinetics than the consolidated product obtained from the atomized powder. However, the HPT product obtained from atomized powder could absorb more hydrogen and showed faster desorption kinetics corresponding to a lower activation energy. These results are discussed by taking into account the effectiveness of the HPT process to refine the grain sizes and differences in the dispersion of fine MgO oxide particles.

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Introduction

A considerable amount of research has been devoted to develop advanced H-storage media in solid-state. As a promising H carrier, magnesium has been considered as an excellent candidate for H-storage mainly due to its light weight, abundance in the earth's crust and high gravimetric storage capacity (7.6 wt% H) [1,2]. In addition, due to their high thermal energy density the Mg-based metal hydride systems are also considered as potential high temperature heat storage media [3,4]. Unfortunately, the sluggish hydriding/dehydriding kinetics, and stable thermodynamic properties [5] leading to higher processing temperatures are the main obstacles for metal-H systems for potential industrial applications.

Significant improvements in the H-sorption kinetics of metal-H systems have been achieved by the use of high-energy-ball-milling (HEBM) [6–12], which were primarily considered to be the effect of nano-metric crystallite/grain size, introduction of lattice defects and increase of reactive surface area. The catalytic action of additions of transition metals or oxides was also considered to be beneficial. In particular, Barkhordarian et al. [8] showed that milling of MgH_2 with 1 mol% of different oxides such as TiO_2 , V_2O_5 or Nb_2O_5 leads to an improvement of magnesium H-sorption kinetics. Aguey-Zinsou et al. [9] considered that the major effect of the M_xO_y oxides was related to the refinement of the size of MgH_2 particles during milling. It was also reported that co-milling of Mg with its own MgO does not modify the structural and thermodynamic properties of MgH_2 but leads to an efficient decrease of the particle size and therefore enhances the H-sorption kinetics [10]. Although the milling route results in considerable improvements in H-sorption properties, it suffers from two major drawbacks. Firstly, it is difficult to avoid agglomeration during milling of Mg or Mg alloys [11]. Secondly, impurities coming from the milling tools and gas adsorption are hard to control and can affect significantly the H-sorption kinetic properties [12].

To avoid these drawbacks, severe plastic deformation (SPD) techniques have been recently used such as equal-channel angular pressing (ECAP) and high pressure torsion (HPT) [13–18]. It was suggested from SPD on pure Mg that the refinement in grain size (i.e. large increase in amount of grain boundaries) played a more effective role than the amount of dislocations for modifying the H-storage properties [13]. Although the compaction of powders under high pressure plus torsional straining has been initiated already in the 90's [19,20], processing routes involving the consolidation of powders using SPD techniques for H-storage materials have received comparatively little attention [21–26]. Kusadome et al. [21] investigated the effect of high-pressure torsion (HPT) on MgNi_2 powders and found that the intermetallic (normally accepted as non-absorber) could absorb 0.1 wt% H even at 100 °C. Leiva et al. [22] have employed several SPD techniques (HPT, cold rolling and forging) on MgH_2 and MgH_2 – Fe powder mixtures and reported that SPD can also reduce crystallite size. Liang and Hout [24] showed that the application of cold rolling on MgH_2 powders could be an efficient processing route to produce nanocrystalline structure for improving H-sorption properties.

A new method – applying HPT on arc plasma evaporated ultrafine grained powder – has been recently introduced to produce bulk Mg-MgO based nanocomposite for H storage [25,26]. The improved H-sorption properties of these bulk composites were mainly attributed to the refinement of the Mg matrix and the dispersion of fine MgO particles formed during intense shearing by HPT [25]. The effect of small amount of Fe and Ni addition was also examined [26]. This new processing route has potentially several advantages. Compared to HEBM, it is less likely to introduce contamination within the heavily deformed material. Also, in comparison with other SPD routes like, for example, ECAP, it is simpler to control and easier to apply on powder.

In this context, in the present study the same processing route was employed with the aim of examining the effect of the nature of the initial powder precursors. To this end, two distinct Mg powder precursors consisting of micro-sized powder particles produced by inert gas atomization and ultrafine powder particles synthesized by an arc plasma evaporation method were employed. The idea of using atomized micro-sized powder instead of the evaporated ultrafine powder lies also in the fact that the production of atomized powder is much cheaper and can be done in more significant quantities.

Experimental

Sample processing

Two Mg powder precursors having different particle sizes were used in the present work for HPT processing. Commercial purity magnesium (99.8%) was gas atomized by SFM SA (Switzerland) to produce a micro-sized Mg powder with a particle size distribution in the range of 10–80 μm . Ultrafine magnesium powder was also produced by arc plasma evaporation/condensation method to generate particle sizes in the range of 50–600 nm. In the latter case, the apparatus was filled with a mixture of 0.70 atm Ar and 0.10 atm H_2 . The details of this procedure are available in Refs. [27,28]. In order to prevent these very fine Mg particles from burning in open air, a two-step passivation procedure was applied: maintaining the powder for 2 h in a mixture of 50 kPa Ar + 10 kPa normal air followed by holding for 12 h in a mixture of 50 kPa Ar + 50 kPa normal air, before opening the evaporation/condensation chamber [27,28].

Fig. 1 shows images illustrating the aspects of the different powders. The atomization process led to fairly rounded particles (see Fig. 1a) having a particle size distribution centered on ~15 μm in the micro-sized powder. Typical SEM image and TEM image of the ultrafine powder are shown in Fig. 1b. They demonstrate that the particle size distribution was essentially in the sub-micrometer range with an average particle size below 300 nm, and that each powder particle corresponded generally to a single grain. The hexagonal shape of some finer powder particles, visible in the TEM image, is due to the hexagonal structure of the Mg phase [25].

These two powders were separately processed by a two-step HPT facility. A schematic diagram of the processing route employed in this study is illustrated in Fig. 2. The

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