

Crystallography and Morphology of (Gd,Y)H₂ Hydride in a Mg-Gd-Y-Al Alloy

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Abstract: Hydrogen can be easily captured by the rare-earth (RE) elements in hydrogen-rich environments, which significantly affect the phase compositions and mechanical performance of Mg-RE based alloys. However, the morphology of RE hydrides and their orientation relationships (ORs) with the Mg matrix have not been well explained. Here, a stable face-centered cubic (FCC) (Gd,Y)H₂ hydride was introduced and uniformly distributed in a Mg-15Gd-2.5Y-1Al alloy after hydrogenation treatment at 500 °C and 2 MPa for 40 h. The plate-like (Gd,Y)H₂ hydride with six variants was identified to exhibit an OR with the magnesium (Mg) matrix, which is $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$, $(10\bar{1}0)_{\text{Mg}}$ 10.5° from $(002)_{(\text{Gd,Y})\text{H}_2}$, $(\bar{1}210)_{\text{Mg}}$ 10.5° from $(020)_{(\text{Gd,Y})\text{H}_2}$. Further crystallographic matching calculations based on the edge-to-edge matching model suggest that such an OR is energetically favorable and provides the actual interface between the RE hydrides and the Mg matrix during precipitation. Our findings offer new insights into the microstructural regulation of Mg alloys in hydrogenation environments.

Keywords: magnesium alloys; hydride; crystallography; morphology



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1. Introduction

Hydrogen has garnered significant attention as a promising alternative to fossil fuels in the emerging energy sector [1]. Metals play a crucial role in the transportation, storage, and utilization of hydrogen [2]. However, hydrogen can significantly reduce the fracture toughness and ductility of metals and thus lead to catastrophic failure, which is known as hydrogen embrittlement [3]. This poses a considerable risk to the widespread and safe application of hydrogen. Although hydrogen embrittlement has been intensively investigated in steel [4,5], aluminum alloys [6,7], and titanium alloys [8,9], the relevant research on the lightest structural metal—magnesium (Mg)—alloys [10,11] is far from sufficient. Although it has been reported that hydrogen segregation at grain boundaries can lead to premature intergranular cracking in Mg alloys [12,13], some recent studies have suggested that the ductility of hydrogen-treated rare-earth (RE) Mg alloys can be significantly improved [14–16]. This is mainly attributed to the high affinity of RE elements for hydrogen and the formation of RE hydrides [17,18]. In other words, hydrogenation treatment of Mg-RE alloys will dramatically change the phase compositions and microstructures. As a secondary phase, the morphology of RE hydrides and their orientation relationships (ORs) with the Mg matrix can significantly affect the mechanical properties of the alloys [19]. However, while some research has focused on the morphology of RE hydrides [20–22], there is still a significant gap in understanding their ORs with the Mg matrix.

In this work, a Mg-Gd-Y-Al alloy was selected to investigate the precipitation behavior of Mg alloys following hydrogen treatment. Gadolinium (Gd) and yttrium (Y) are the most commonly used RE elements in Mg alloys due to their high solubility, which results in remarkable solid solution strengthening and precipitation strengthening effects [23,24]. They were also reported to readily combine with hydrogen to form dihydride [17,20]. To gain higher strength and corrosion resistance, Al was added to the alloy to refine the grain size and form a passive film [25–27]. The microstructure of this Mg-Gd-Y-Al alloy before and after hydrogenation treatment was studied. The morphological crystallography of RE hydrides was comprehensively characterized by X-ray diffraction (XRD), electron backscattered diffraction (EBSD), and transmission electron microscopy (TEM) and rationalized by contemporary interphase crystallographic models.

2. Materials and Methods

2.1. Materials

The Mg-Gd-Y-Al alloy was prepared with pure Mg (99.9%), Mg-30Gd master alloy (99.9%), Mg-30Y master alloy (99.9%), and pure Al (99.9%) using a gravity casting method under the protection of CO₂ and SF₆. The chemical composition of the alloy was measured to be Mg-15Gd-2.5Y-1Al (wt.%) using an inductively coupled plasma optical emission spectrometer (Avio 500, Perkin Elmer, Waltham, MA, USA). Hydrogenation experiments were conducted using an Ultra High Pressure Gas Sorption Analyzer (BSD-PHU, Beishide Company, Beijing, China) with high-purity H₂ (99.999%) as the adsorption gas. Cubic samples with dimensions of 10 mm * 10 mm * 10 mm were cut from the casting alloy and then placed in the hydrogen adsorption chamber. The samples were treated with hydrogen permeation conditions at 500 °C and 2 MPa for 40 h.

2.2. Methods

The phase composition of the alloy before and after hydrogenation was analyzed by XRD and EBSD. The EBSD sample was sequentially polished using 320#, 800#, and 2000# grit sandpaper, followed by polishing with 3 μm, 1 μm, and 0.05 μm polishing suspensions. The ORs between the hydrides and the Mg matrix were determined by EBSD and TEM (JEOL ARM200F, JEOL, Tokyo Metropolis, Japan). The sample was thinned to ~60 μm using 1200#, 3000#, and 7000# grit sandpaper. Double jet polishing was carried out using an electrolytic double jet polisher with a 4 vol.% perchloric acid in alcohol solution at a working voltage of 40 V. Subsequently, the sample was further thinned for 20 min using an ion thinning instrument under conditions of ±2.5° tilt and 1 keV energy. The crystallographic matching between the Mg matrix and the RE hydrides was analyzed using the edge-to-edge matching (E2EM) model, which has been utilized to explain and predict the ORs between precipitates and matrices in numerous alloy systems [28]. The fundamental principles of this model have been previously described [29]. By inputting the crystal structures and lattice parameters of both the Mg matrix and the RE hydrides, the energetically favorable ORs can be identified.

3. Results and Discussion

Figure 1 presents the XRD results of the Mg-Gd-Y-Al alloy before and after hydrogenation. The as-cast alloy contains two types of eutectic phases, namely Mg₂₄(Gd, Y)₅ and Al₂(Gd, Y). After hydrogenation, both of these second phases disappeared, while two types of hydrides, i.e., MgH₂ and (Gd, Y)H₂, were observed. Figure 2 summarizes the phase maps, inverse pole figure (IPF), and pole figure (PF) of the alloy before and after hydrogenation. As shown in Figure 2a, Mg₂₄(Gd, Y)₅, indicated in cyan color, mainly distributes along the grain boundaries or at the grain boundary junctions. The Al₂(Gd, Y)

phase is randomly dispersed in the intergranular or intragranular regions. Figure 2b,c show the phase composition after hydrogenation. Consistent with the XRD results, the original second phases in the as-cast alloy disappeared, while MgH_2 and $(\text{Gd}, \text{Y})\text{H}_2$ formed. It can be seen that most of the hydrides are $(\text{Gd}, \text{Y})\text{H}_2$ and very few are MgH_2 (as indicated by the white circle in Figure 2c). This suggests that the hydrogenation of the Mg matrix is greatly inhibited under the present hydrogenation conditions and most of the hydrogen is bonded to the RE elements. Figure 2c,d show the magnified phase map and IPF of the region highlighted by the white box in Figure 2b, respectively. As highlighted in the black box, a large number of plate-like $(\text{Gd}, \text{Y})\text{H}_2$ phases precipitated from the Mg matrix. The $\{0001\}$ -Mg PF and $\{001\}$ -($\text{Gd}, \text{Y})\text{H}_2$ PF in Figure 2e suggest that the $(\text{Gd}, \text{Y})\text{H}_2$ phase shows a specific OR with the Mg matrix with six different variants, and these six variants are sequentially numbered from 1 to 6. All these variants share a common (001) pole point, which coincides with the projection of the (0001) pole point of the Mg matrix. Details of the OR will be discussed later. In addition to intragranular precipitated $(\text{Gd}, \text{Y})\text{H}_2$, continuous distributions of the $(\text{Gd}, \text{Y})\text{H}_2$ phase along the grain boundaries and hydride clusters were also observed. It is likely that the original $\text{Mg}_{24}(\text{Gd}, \text{Y})_5$ and $\text{Al}_2(\text{Gd}, \text{Y})$ served as precursors for the formation of these $(\text{Gd}, \text{Y})\text{H}_2$ precipitates.

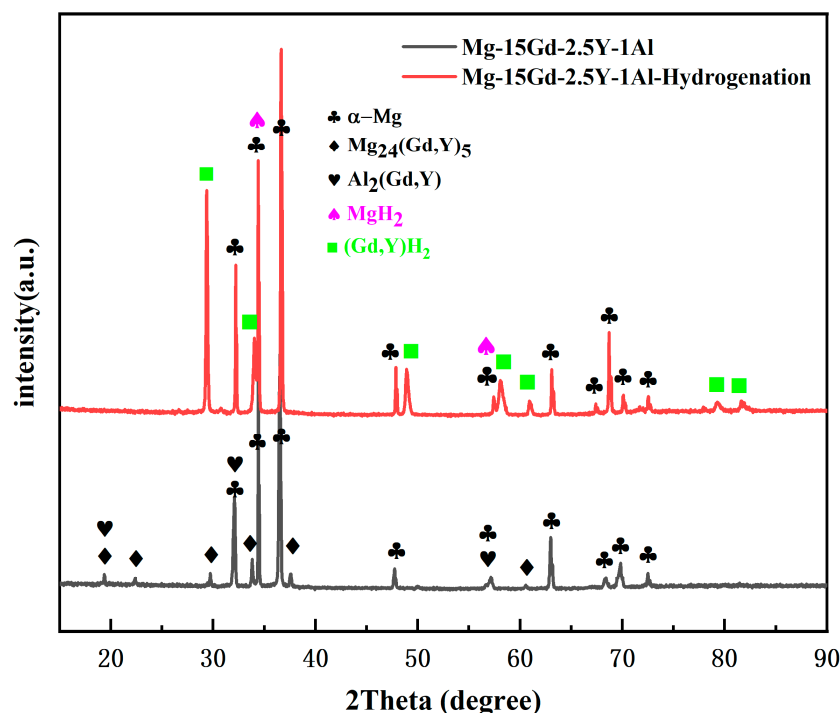


Figure 1. The XRD results of the alloy before and after hydrogenation.

The OR between the intragranular plate-like $(\text{Gd}, \text{Y})\text{H}_2$ and the Mg matrix was further investigated by TEM. Figure 3a shows the bright-field (BF) TEM image of the sample observed along the $[0001]_{\text{Mg}}$ direction. The average thickness of the plate-like $(\text{Gd}, \text{Y})\text{H}_2$ is approximately 100 nm. As indicated by the blue lines, six different variants can be observed within a single grain. Herein, three of these variants are selected, as boxed in red, and the corresponding Selective Area Electron Diffraction (SAED) patterns are given in Figure 3b–d. As can be seen from Figure 3b, the four diffraction spots closest to the transmitted beam connected by a green square confirm that the FCC-structured $(\text{Gd}, \text{Y})\text{H}_2$ phase is oriented along the $[001]$ direction. As indicated by the yellow dashed line, the (002) plane of $(\text{Gd}, \text{Y})\text{H}_2$ and the $(0\bar{1}10)$ plane of the Mg matrix are misaligned by an angle of 10.1° . Thus, the OR can be expressed as follows: $[0001]_{\text{Mg}} // [001]_{(\text{Gd}, \text{Y})\text{H}_2}$, $(0\bar{1}10)_{\text{Mg}} 10.1^\circ$ from $(002)_{(\text{Gd}, \text{Y})\text{H}_2}$.

$(2\bar{1}\bar{1}0)_{\text{Mg}}$ 10.1° from $(020)_{(\text{Gd,Y})\text{H}_2}$. Similar to that in Figure 3b, the OR in Figure 3c can be represented as $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$, $(10\bar{1}0)_{\text{Mg}}$ 10.5° from $(002)_{(\text{Gd,Y})\text{H}_2}$, $(\bar{1}2\bar{1}0)_{\text{Mg}}$ 10.5° from $(020)_{(\text{Gd,Y})\text{H}_2}$. The OR in Figure 3d can be expressed as $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$, $(1\bar{1}00)_{\text{Mg}}$ 10.7° from $(002)_{(\text{Gd,Y})\text{H}_2}$, $(\bar{1}\bar{1}20)_{\text{Mg}}$ 10.7° from $(020)_{(\text{Gd,Y})\text{H}_2}$. All these three ORs are essentially equivalent within the experimental uncertainty. Because there are three equivalent $\{10\bar{1}0\}_{\text{Mg}}$ planes and the diffraction spot of the $\{002\}_{(\text{Gd,Y})\text{H}_2}$ plane can stay on either side of $\{10\bar{1}0\}_{\text{Mg}}$ with the same misalignment angle, this OR has six equivalent variants, which explains the six traces in the BF image (Figure 3a) as well as the six pairs of $\{010\}$ pole points in Figure 2e.

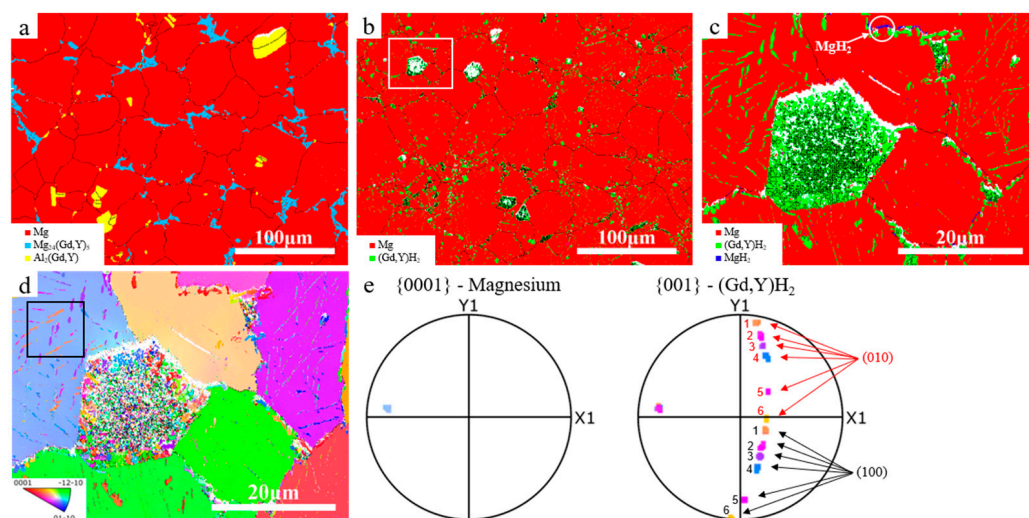


Figure 2. EBSD analysis of samples before and after hydrogenation. (a) Phase map before hydrogenation. (b,c) Phase maps after hydrogenation. (d) IPF of the region highlighted by the white box in (b). (e) PF of the area within the black box in (d).

The occurrence of this unusual OR between hexagonal close-packed (HCP, the Mg matrix) and face-centered cubic (FCC, the plate-like (Gd,Y)H₂ precipitates) can be well explained from a crystallographic matching perspective. In the HCP/FCC system, the atomic row mismatch along the close-packed directions $[0001]_{\text{Mg}}$ and $[001]_{(\text{Gd,Y})\text{H}_2}$ is only 0.85%. Thus, this is considered to be a pair of matching rows owing to the low interatomic mismatch along the row. As required by the E2EM model, this pair of matching rows must coincide or match at the interface to minimize the interface energy [29]. Figure 4a presents the simulated superimposed diffraction pattern of the Mg matrix and (Gd,Y)H₂ along the $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$ direction based on the E2EM model. In this case, the pair of matching planes that carry this matching pair of rows to the interface is $(0\bar{1}\bar{1}0)_{\text{Mg}}$ and $(002)_{(\text{Gd,Y})\text{H}_2}$ with an interplanar spacing misfit of 5.98%. To maintain atomic row alignment at the interface, these two matching planes must rotate by a certain degree relative to each other about the matching direction [30]. The calculated misalignment angle between $(0\bar{1}\bar{1}0)_{\text{Mg}}$ and $(002)_{(\text{Gd,Y})\text{H}_2}$ is 9.5° according to the second $\Delta\mathbf{g}$ parallelism rule, i.e., $\Delta\mathbf{g}_1 // \Delta\mathbf{g}_2$, where $\Delta\mathbf{g}_1 = \mathbf{g}_{1\bar{2}\bar{1}0} - \mathbf{g}_{020}$ and $\Delta\mathbf{g}_2 = \mathbf{g}_{10\bar{1}0} - \mathbf{g}_{002}$. It should be noted that there exists a $\sim 1^\circ$ discrepancy between the experimentally observed and simulated ORs in terms of the angle between the $(0\bar{1}\bar{1}0)_{\text{Mg}}$ and $(002)_{(\text{Gd,Y})\text{H}_2}$ planes. This may be attributed to the difference between the nominal lattice constants of (Gd,Y)H₂ used for crystallographic matching calculation and the actual lattice constants, due to the complex structure of the RE hydrides [17,18,31,32]. In practice, the atomic ratio of (Gd,Y) and H in the RE hydrides can vary between 1.9 and 2.3 [17], potentially affecting the lattice constants and, consequently, the OR.

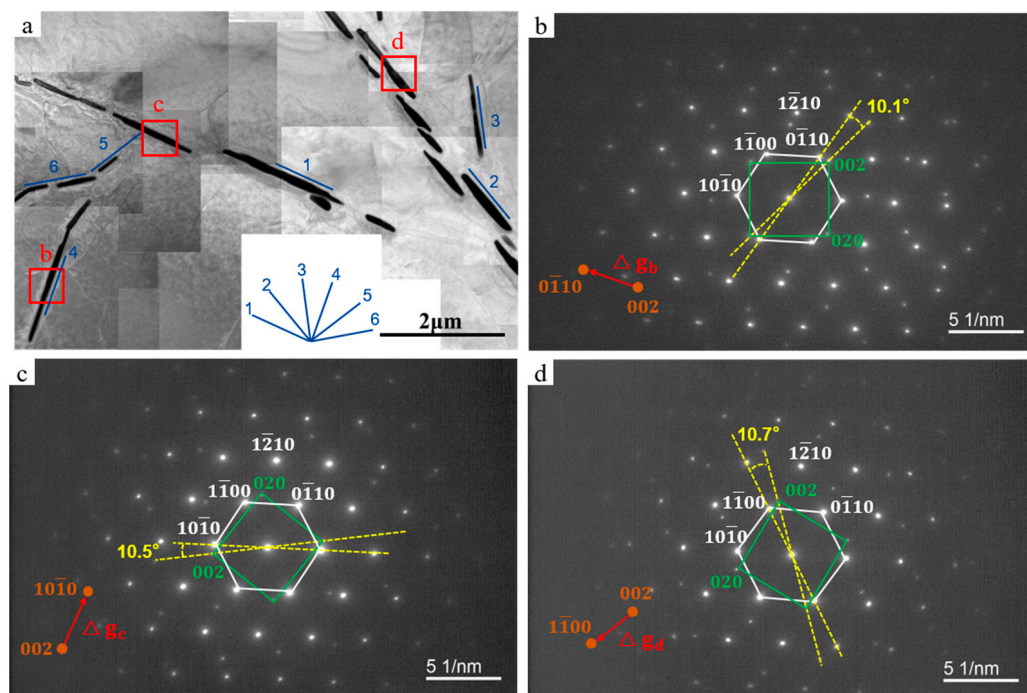


Figure 3. Intragranular plate-like hydride precipitates analyzed by TEM. (a) A mosaic of bright-field images showing the six variants of hydrides sharing the same OR. Number these six variants from NO. 1 to 6 (blue Arabic numerals). (b–d) Superimposed SAED patterns of the Mg matrix and RE hydrides in the vicinity of three variants (No. 1, 2, and 4, respectively) as indicated in (a). The specific positions are shown in the red box in (a), with the corresponding red letters at the top corresponding to (b–d). Note the Δg vectors are defined as the difference of $g\{10\bar{1}0\}$ and $g\{002\}$, as shown in the inset of (b–d).

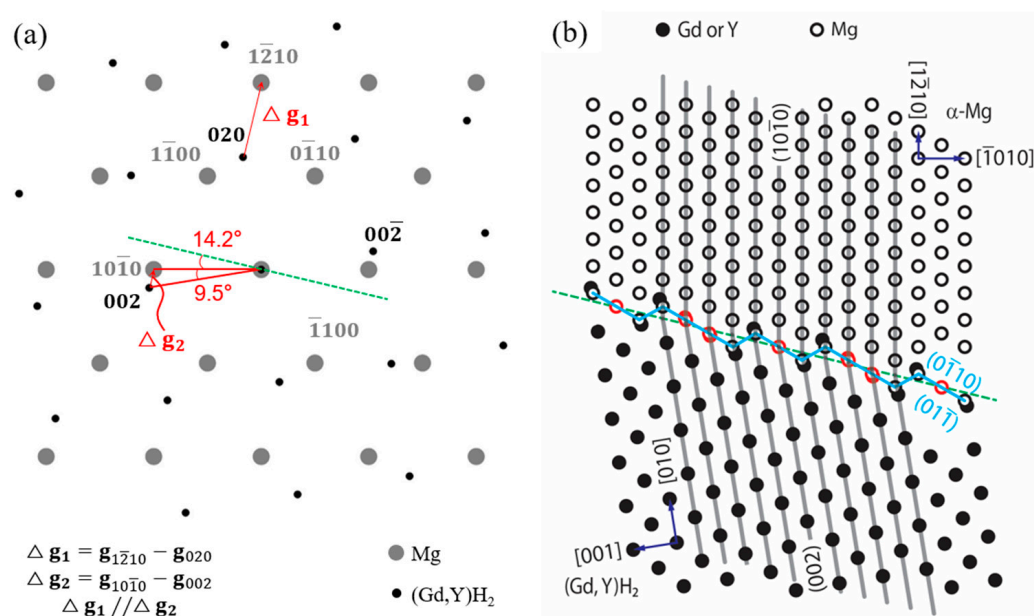


Figure 4. (a) Simulated diffraction patterns of intragranular plate-like hydrides and their Mg matrix at the OR suggested by the second Δg parallelism rule. The green dashed line indicates the trace of the habit plane. The large gray circle denotes the Mg matrix, while the small black circles represent $(\text{Gd}, \text{Y})\text{H}_2$. (b) Local interfacial structure showing the “edge-to-edge” matching manner between $(10\bar{1}0)_{\text{Mg}}$ and $(002)_{(\text{Gd}, \text{Y})\text{H}_2}$ across the interface at the predicted OR. The regularly distributed red circles are well-matched atoms along the stepped interface with the terrace plane of $(0\bar{1}10)_{\text{Mg}}$ and $(01\bar{1})_{(\text{Gd}, \text{Y})\text{H}_2}$.

The energetically favorable interface orientation can also be calculated, defined by this pair of parallel $\Delta\mathbf{g}$ vectors, which can be expressed as $(0.93\ 0.36\ \overline{1.30}\ 0.00)_{\text{Mg}} // (0.00\ \overline{0.40}\ 0.92)_{(\text{Gd,Y})\text{H}_2}$. The green dashed line in Figure 4a represents the interface trace line perpendicular to the parallel $\Delta\mathbf{g}$ vectors, which is also consistent with the actual interface (see the trace of precipitate No. 1, or (c) in Figure 3a) between the RE hydrides and the Mg matrix during precipitation. Figure 4b shows a simulated atomic structure of this high-index interface observed along the $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$ direction. The projections of atomic rows in the Mg and (Gd, Y)H₂ lattices are represented by open circles and solid circles, respectively. The atomic species within a single lattice are not distinguished. For brevity, only the terraces and steps in the Mg lattice are illustrated by solid lines. The terrace plane is the $(0\overline{1}10)_{\text{Mg}}$ plane on the Mg side and the $(01\overline{1})_{(\text{Gd,Y})\text{H}_2}$ plane on the (Gd, Y)H₂ side. There is an angular discrepancy of 5.5° between these two planes, which allows the matching of terrace planes along the edges across the interface. The primary matching planes, $[100]_{\text{Mg}}$ and $[002]_{(\text{Gd,Y})\text{H}_2}$, are highlighted by thick, gray solid lines, and they match each other in an “edge-to-edge” manner along the trace of the predicted interface. This stepped interface structure effectively eliminates all lattice mismatches between adjacent rows along the matching direction at the interface, thereby minimizing the structural component of the interfacial energy [33].

4. Conclusions

In summary, the microstructure evolution of a Mg-Gd-Y-Al alloy after hydrogenation treatment was investigated in detail in this work. The initial eutectic phases, including Mg₂₄(Gd, Y)₅ and Al₂(Gd, Y), present in the as-cast alloy were found to have completely disappeared after hydrogenation, confirming their full conversion through hydrogenation. Massive plate-like (Gd, Y)H₂ precipitated from the matrix, while the formation of MgH₂ was almost suppressed. The plate-like (Gd, Y)H₂ exhibited a specific OR with the Mg matrix with six equivalent variants, which can be written as $[0001]_{\text{Mg}} // [001]_{(\text{Gd,Y})\text{H}_2}$, $(10\overline{1}0)_{\text{Mg}} \sim 10^\circ$ from $(002)_{(\text{Gd,Y})\text{H}_2}$, $(1\overline{2}10)_{\text{Mg}} \sim 10^\circ$ from $(020)_{(\text{Gd,Y})\text{H}_2}$. Further crystallographic matching calculations suggest that such an OR is energetically favorable. These findings contribute to understanding the precipitation behavior of RE hydrides and improving the mechanical properties of the material by controlling the microstructure of the precipitated phases.

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