Exploring the Hydrogen-Induced Amorphization and Hydrogen Storage Reversibility of Y(Sc)_{0.95}Ni_{2} Laves Phase Compounds

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Abstract: Rare-earth-based AB_{2}-type compounds with Laves phase structure are readily subject to hydrogen-induced amorphization and disproportionation upon hydrogenation. In this work, we conducted the Sc alloying on Y_{0.95}Ni_{2} to improve its hydrogen storage properties. The results show that the amorphization degree of Y_{0.95}Ni_{2} deepens with the increasing hydrogenation time, pressure, and temperature. The Y(Sc)_{0.95}Ni_{2} ternary compounds show a significant improvement in reversibility and dehydriding thermodynamics due to the reduced atomic radius ratio R_{A}/R_{B} and cell volume. Hydrogen-induced amorphization is fully eliminated in the Y_{0.25}Sc_{0.7}Ni_{2}. The Y_{0.25}Sc_{0.7}Ni_{2} delivers a reversible hydrogen storage capacity of 0.94 wt.% and the dissociation pressure of 0.095 MPa at the minimum dehydriding temperature of 100 °C.

Keywords: hydrogen storage alloys; hydrogen-induced amorphization; Sc substitution; reversibility

1. Introduction

Numerous intermetallic compounds in the formula of AB_{n}, in which A is the strong hydride-forming elements and B is weak hydride-forming elements, have been considered to be a potential hydrogen storage medium for either gaseous or electrochemical applications [1–6]. Among them, AB_{2}-type intermetallic compounds possess higher theoretical specific capacity than the most widely used AB_{5}-type rare-earth compounds. Zr-based and Ti-based compounds with cubic MgCu_{2}-type (C15) or hexagonal MgZn_{2}-type (C14) structure could store more than 2.0 wt.% hydrogen [7–9]. However, the rare-earth-based AB_{2}-type compounds have rarely been considered for reversible hydrogen storage because of the hydrogen-induced amorphization (HIA) and phase disproportionation (HID) upon hydrogenation [10–17]. For example, the YFe_{2} with cubic C15 structure would experience four steps of structural change in the hydrogenation: the formation of crystalline hydride YFe_{2}H_{x}, the transformation of crystalline YFe_{2}H_{x} to non-crystalline YFe_{2}H_{x}, precipitation of YH_{3} from non-crystalline hydride, and the decomposition into YH_{3} and α-Fe [18].

The HIA and HID mechanisms of several representative rare-earth-based AB_{2}-type compounds have been comprehensively investigated in early studies by Aoki et al. [19,20]. The hydrogenation pressure, time, and temperature dependence has been demonstrated. For example, the structural change of DyNi_{2} during hydrogen absorption is dependent on the pressure. The decomposition phases DyNi_{6} and DyH_{3} were finally obtained above 2.0 MPa hydrogen [21]. The occurrence of HIA and HID means the structural instability of AB_{2}-type compounds, especially as hydrogen atoms enter the tetrahedral interstices and cause the lattice expansion. The structural stability of rare-earth-based AB_{2}-type compounds is closely related to the atomic radius ratio R_{A}/R_{B} of the constituting A and B elements. The ideal R_{A}/R_{B} value for the cubic Laves phase structure is 1.225 [22], and the larger the R_{A}/R_{B} deviation from the value of 1.225, the more unstable Laves phase structure. Aoki et al. proposed a critical R_{A}/R_{B} value of 1.37 as the judgment factor of HIA by summarizing a large variety of AB_{2}-type hydrogen storage compounds [20].
Since rare-earth elements possess a relatively large atomic radius, most rare-earth-based AB₂-type compounds have a \( \text{R}_A / \text{R}_B \) value exceeding 1.37 and, thus, are readily subject to HIA.

Rare-earth-based compounds form the largest subset of the AB₂-type Laves phase compounds [23], and their potential for hydrogen storage is worthy of more exploration to overcome the HIA problem by compositional and structural modification. Our previous studies show that the B-side substitution with Al and the A-side alloying with Zr could effectively inhibit the HIA and achieve reversible hydrogen storage [24–27]. The \( \text{YFe}_{1.7}\text{Al}_{0.3} \) delivers a reversible hydrogen storage capacity of 1.38 wt.%, but the complete hydrogen desorption must be conducted in a dynamic vacuum at 200 °C [24]. The Zr alloying could realize the reversible hydrogen absorption with the elevated dissociation pressure of \( \text{YFe}_2 \) because of the reduced cell volume [25]. Zhang et al. [28] reported that the pseudo-binary \( \text{Sm}_{1.25}\text{Mg}_{0.75}\text{Ni}_4 \) compound could reversibly absorb and desorb ca. 0.95 wt.% hydrogen without the occurrence of HIA at 25 °C.

Regarding the \( \text{Y}_{0.95}\text{Ni}_2 \) compound with a C15 superlattice structure, the crystalline hydride \( \text{Y}_{0.95}\text{Ni}_2\text{H}_{2.6} \) with a capacity of 1.27 wt.% has been reported, but further increasing the hydrogen capacity would result in amorphization [29]. Zhang et al. reported that the \( \text{Y}_{0.95}\text{Ni}_2 \) experienced gradual amorphization during absorption/desorption cycling under gaseous hydrogen at ambient temperature [30]. In this work, we conducted the Sc alloying on \( \text{Y}_{0.95}\text{Ni}_2 \) to suppress the HIA because the Sc element has a relatively smaller atomic radius (1.66 Å) than Y (1.78 Å), and, thus, the \( \text{R}_A / \text{R}_B \) value could be decreased. The effect of Sc content on the microstructure and hydrogen storage properties of Y–Sc–Ni compounds have been investigated.

2. Materials and Methods

Commercial Y, Sc, and Ni ingots with a purity of 99.9% were used as the starting materials. \( \text{Y}_{0.95-x}\text{Sc}_x\text{Ni}_2 \) (x = 0, 0.1, 0.3, 0.5, 0.6, 0.7) alloys were prepared by arc melting under a high purity Argon atmosphere with Y, Sc, and Ni bulks in proper proportion. All samples were remelted four to five times and annealed at 950 °C for 4 days to obtain homogenous composition and structure. The alloys were crushed into powder and sieved by a 200-mesh (~74 um) sieve in the glove box for further measurement.

The composition and microstructure were observed using a scanning electron microscope (SEM, Zeiss Supra 40/VP, Carl Zeiss AG, Jena, Germany) equipped with an energy-dispersive spectrometer (EDS). X-ray diffraction (XRD) measurements were conducted using a PANalytical Empyrean with Cu Ka radiation (\( \lambda = 1.5406 \) Å, PANalytical B.V., Almelo, The Netherlands). Rietveld refinements were conducted by using the Highscore Plus 4.5 software (PANalytical B.V.).

To investigate hydrogen-induced amorphization, the alloy powder was loaded into stainless steel reactors in an Argon-filled glove box. Then, the alloy powder was subjected to a hydrogenation reaction under different hydrogenation conditions: 3 MPa \( \text{H}_2 \)-100 °C-2 h, 3 MPa \( \text{H}_2 \)-100 °C-24 h, 3 MPa \( \text{H}_2 \)-200 °C-2 h, and 5 MPa \( \text{H}_2 \)-100 °C-24 h. The hydrogen desorption and crystallization processes of hydrogenated samples were monitored by differential scanning calorimetry (DSC, Setaram SENSYS Evolution, SETARAM Instrumentation, Lyon, France) at a heating rate of 5 °C/min.

The measurement of hydrogen storage properties was conducted on an automatic Sieverts-type apparatus (AMC HP2000, Advanced Materials Corporation, Pittsburgh, America). About a 1.3 g sample was put into the sample holder, the hydriding, and dehydriding pressure-composition isotherms (PCI) of \( \text{Y}_{0.25}\text{Sc}_{0.7}\text{Ni}_2 \) compound were measured at different temperature.

3. Results

3.1. Structure of \( \text{Y(Sc)}_{0.95}\text{Ni}_2 \) Compounds

Figure 1 shows the XRD patterns of as-annealed \( \text{Y}_{0.95-x}\text{Sc}_x\text{Ni}_2 \) (x = 0, 0.1, 0.3, 0.5, 0.6, 0.7) compounds. The structural parameters of all compounds are refined by the Highscore
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Plus 4.5 software and summarized in Table 1. For the compounds with the Sc content of 0, 0.1, and 0.3, in addition to the strong Bragg peaks of cubic C15 Laves phase (MgCu2-type structure, space group Fd3m), some extra weak Bragg peaks indicate the formation of the C15 superstructure (C15s) with the space group of Fd3m. The C15s structure of Y0.95Ni2 has a doubled lattice constant of 14.35 Å compared with the C15 structure. The structural refinement by Latroche et al. shows that most atoms in the C15s structure are shifted from the ideal atomic positions expected from the C15 structure [31]. It is noted that the Y vacancies in the 4a site could stabilize the Laves phase structure of nonstoichiometric Y0.95Ni2 with a large RA/ RB value of 1.424 [29]. Since the Sc content is larger than 0.3, the weak peaks of the superstructure disappear, implying the transformation from the C15s superstructure to the C15 structure. This transformation should be related to the decrease of the RY/RNi value due to Sc substitution, which results in the decrease of the number of ordered Y vacancies [31]. The Rietveld refinement results indicate that the 4a site occupancy of the Y vacancy for Y0.85Sc0.1Ni2 is 0.421, which decreases to almost zero for the Y0.65Sc0.3Ni2. The Y0.25Sc0.7Ni2 has an RA/ RB value of 1.353, which is much lower than that of the Y0.95Ni2. Therefore, the ScNi2 phase with the C15 structure is identified for the Y0.25Sc0.7Ni2 [32]. EDS analysis (Figure 2) shows that the representative compound Y0.25Sc0.7Ni2 has a homogeneous microstructure and distribution of Y and Sc elements. Further, the EDS compositional analysis of each Y–Sc–Ni compound is highly consistent with the designed ones, as shown in Table 1.

![Figure 1. XRD patterns of as-annealed Y-Sc-Ni compounds.](image)

Table 1. Structural parameters of Y0.95−xScxNi2 compounds.

| Nominal Composition | EDS Composition | Space Group | Crystal Structure | Lattice Constant, a/Å | *RA/RB | HIA |
|--------------------|-----------------|-------------|-------------------|-----------------------|--------|-----|
| Y0.95Ni2.00        | Y0.96(1)Ni2.00(3) | Fd3m        | C15s              | 14.35                 | 1.424  | Yes |
| Y0.85Sc0.10Ni2.00  | Y0.84(1)Sc0.11(1)Ni2.00(3) | Fd3m        | C15s              | 14.29                 | 1.414  | Yes |
| Y0.65Sc0.30Ni2.00  | Y0.66(1)Sc0.31(1)Ni2.00(3) | Fd3m        | C15s              | 14.15                 | 1.394  | partial |
| Y0.45Sc0.50Ni2.00  | Y0.46(1)Sc0.50(1)Ni2.00(3) | Fd3m        | C15               | 7.03                  | 1.373  | partial |
| Y0.25Sc0.70Ni2.00  | Y0.36(1)Sc0.69(1)Ni2.00(3) | Fd3m        | C15               | 7.01                  | 1.363  | partial |

*RA = RY × \frac{0.95}{0.85} + RSc × \frac{0.10}{0.25}, RB = RNi = 1.25 Å, RY = 1.78 Å, RSc = 1.66 Å.
3.2. Hydrogen-Induced Amorphization of Y$_{0.95}$Ni$_2$

The effect of hydrogenation temperature, time, and pressure on the HIA of Y$_{0.95}$Ni$_2$ is investigated. Figure 3 compares the structures of Y$_{0.95}$Ni$_2$ hydrogenated under different conditions with the as-annealed Y$_{0.95}$Ni$_2$. After hydrogenation at 100 °C-3 MPa-2 h, Figure 3b shows that all Bragg peaks shift toward the low angle direction, indicating the formation of crystalline hydride of Y$_{0.95}$Ni$_2$. The interstitial hydride also adopts the same C15 structure. As shown in Figure 3c,d there exists a broad scattering peak at 30° to 45° in addition to the diffractions of crystalline hydride, indicating the formation of amorphous phase at an elevated temperature of 200 °C or prolonged time of 24 h. It is noted that the present Y$_2$O$_3$ phase may be related to the partial oxidization of Y during the arc-melting or hydrogenation. When hydrogenated at 100 °C-5 MPa-24 h, the hydrogenated product consists of the major amorphous phase, indicating full transformation from crystalline hydride to non-crystalline hydride under the elevated hydrogenation pressure. Hence, it is assumed that the increased hydrogen storage capacity due to the increased hydrogenation temperature, pressure, and time results in the destruction of the Laves phase structure of Y$_{0.95}$Ni$_2$ hydride, which is in line with other rare-earth RENi$_2$ compounds [14].

![Figure 2. EDS elemental mapping (a–d) of as-annealed Y$_{0.25}$Sc$_{0.7}$Ni$_2$.](image)

![Figure 3. XRD patterns of Y$_{0.95}$Ni$_2$ after hydrogenation under different temperatures, hydrogen pressure, and times. (a) Initial Y$_{0.95}$Ni$_2$; (b) 100 °C-3 MPa-2 h; (c) 100 °C-3 MPa-2 h; (d) 100 °C-3 MPa-24 h; (e) 100 °C-5 MPa-24 h.](image)
3.3. Hydrogen-Induced Amorphization of Y–Sc–Ni Compounds

Figure 4 shows the XRD patterns of $Y_{0.95-x}Sc_xNi_2$ ($x = 0.1, 0.3, 0.5, 0.6, 0.7$) compounds after hydrogenation at 100 °C-5 MPa-24 h. Like the $Y_{0.95}Ni_2$, the full HIA occurs for the $Y_{0.85}Sc_{0.1}Ni_2$. With the increasing Y content of 0.3–0.6, the Bragg peaks of crystalline hydride with a C15 structure become intensifying, indicating that the HIA is partially suppressed due to the Sc alloying. The absence of the broadening scattering peak and the presence of C15 diffractions indicate that the HIA is fully eliminated in the hydrogenation of $Y_{0.25}Sc_{0.7}Ni_2$. As shown in Table 1, the $R_A/R_B$ value of Y–Sc–Ni compounds decreases with increasing Sc content, being 1.353 for the $Y_{0.25}Sc_{0.7}Ni_2$. This result implies that the Sc alloying could improve the structural stability of $Y_{0.95}Ni_2$, which should be the structural reason for eliminating HIA.

![Figure 4. XRD patterns of Y–Sc–Ni compounds after hydrogenation at 100 °C-5 MPa-24 h.](image)

Figure 5a further shows the DSC profiles of Y–Sc–Ni compounds hydrogenated under 100 °C-5 MPa-24 h. The XRD patterns of DSC samples at specific temperatures are shown in Figure 5b. As shown in Figure 5a, a rather broad endothermic peak is followed by a sharp exothermic peak, which is attributed to the dehydrogenation of non-crystalline hydride and the crystallization of non-crystalline hydride, respectively [33]. This assumption is further confirmed by XRD. At the T1 temperature of the DSC curve of the $Y_{0.95}Ni_2$, no crystalline phase exists in addition to the $Y_2O_3$. At the T2 temperature, the presence of $YH_2$ and $YNi_3$ phases indicates the crystallization of non-crystalline hydride and subsequent decomposition. Similar DSC results are also reported for the GdNi$_2$ [34], SmNi$_2$ [35], and YNi$_2$ [36] compounds. With the increasing Sc content, the broad endothermic peak contains a sharp part with an increasing shaded area. The broad endothermic peak below the broken line still corresponds to the hydrogen desorption from noncrystalline hydride, while the sharp endothermic peak with a shaded area is attributed to hydrogen release from the crystalline hydride [33]. In addition, the exothermic peak area and the peak temperature decrease with increasing Sc content. XRD patterns of DSC samples at T3, T4, T5, and T6 also show the weak peaks of $YH_2$ and $YNi_3$ phases, indicating that the amount of non-crystalline hydride decreases with increasing Sc content. For the hydrogenated $Y_{0.25}Sc_{0.7}Ni_2$, the only sizeable endothermic peak in the DSC curve further verifies the elimination of HIA.
3.4. Hydrogen Storage Properties of Y_{0.25}Sc_{0.7}Ni_{2}

Figure 6a displays the hydriding and dehydriding PCI curves of Y_{0.25}Sc_{0.7}Ni_{2} at different temperatures. There exists a clear plateau region in the hydriding curve, but the dehydriding curve starts to slope. At the midpoint of the PCI curve, the hydriding and dehydriding equilibrium pressures are determined to calculate the enthalpy change ΔH and entropy change ΔS, and the fitted van’t Hoff plots are displayed in Figure 6b. The derived dehydriding ΔH and ΔS for the Y_{0.25}Sc_{0.7}Ni_{2} are 35.59 kJ/mol·H₂ and 93.83 J/K/mol·H₂, respectively. In addition, the Y_{0.25}Sc_{0.7}Ni_{2} exhibits a reversible hydrogen storage capacity of 0.94 wt.% at 100 °C, corresponding to the hydride formula Y_{0.25}Sc_{0.7}Ni_{2}H_{2.92}. However, a small amount of hydrogen could not be released at 80 °C, which should be related to the low dissociation pressure. It is strange that, in the present work, the hydride of Y_{0.25}Sc_{0.7}Ni_{2} has a lower capacity than the Y_{0.95}Ni_{2}D_{2.6} in Reference [29], which may be related to the increased equilibrium hydrogen pressure at a higher testing temperature of 100 °C. It is noted that the Y_{0.95}Ni_{2}D_{2.6} was determined at ambient temperature and deuterium pressure. As shown in the hydriding kinetic curves at 40 °C in Figure S1, the hydrogen absorption capacity increases and then decreases with Sc content. The Y_{0.65}Sc_{0.3}Ni_{2} delivers a maximum hydrogen absorption capacity of 1.53 wt. %, corresponding to the hydride formula Y_{0.65}Sc_{0.3}Ni_{2}H_{2.93}. It is noted that the minimum dehydrogenation temperature of the Y_{0.25}Sc_{0.7}Ni_{2} is much lower than that of Y(Zr)Fe_{2} compounds [24–27]. Therefore, in addition to the improvement in the reversibility due to the reduced R_{A}/R_{B}, the Sc alloying also shows a significant promoting effect in the hydrogen sorption equilibrium pressure because of the reduced lattice constant.
4. Conclusions

The gaseous hydrogen storage reversibility of Y(Sc)_{0.95}Ni_{2} compounds with different Sc contents has been investigated. The hydrogen-induced amorphization of Y_{0.95}Ni_{2} is dependent on the hydrogenation pressure, time, and temperature. The Sc alloying could improve structural stability and suppress the hydrogen-induced amorphization. Fully reversible hydrogen storage has been achieved for the Y_{0.25}Sc_{0.7}Ni_{2}, with a reversible capacity of 0.94 wt.% and dissociation pressure of 0.095 MPa at the minimum dehydrogenation temperature of 100 °C. Our work demonstrates the feasibility of rare-earth-based AB_{2}-type compounds for reversible hydrogen storage applications.
Supplementary Materials: The following are available online at https://www.mdpi.com/1996-1944/14/2/276/s1, Figure S1: Hydriding kinetic curves of Y-Sc-Ni compounds with different Sc contents at 40 °C.

Author Contributions: Data curation, S.Z. and J.L. Formal analysis, S.Z. Funding acquisition, H.W. Investigation, S.Z. Methodology, J.L. Project administration, H.W. Resources, H.W. Supervision, J.L. Writing—original draft, S.Z. Writing—review & editing, H.W. All authors have read and agreed to the published version of the manuscript.

Funding: This work was financially supported by the National Key R&D Program of China (Grant No. 2019YFB1502105), the National Natural Science Foundation of China (Grant Nos. U1601212, 51871098), the Fund for Innovative Research Groups of the National Natural Science Foundation of China (Grant No. 51621001), and the Natural Science Foundation of Guangdong Province (2016A030312011).

Institutional Review Board Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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