Postprint: Magnetocaloric Effect of La0.9Ce0.1Fe11.44Si1.56Hy Alloy and Its Powder-Bonded Bulk

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Date: 2023-03-19T00:00:00+00:00

Abstract

The La0.9Ce0.1Fe11.44Si1.56 alloy was subjected to saturated hydrogen absorption, followed by 3 h hydrogen desorption treatment at different desorption temperatures (Td=200~250 ℃) to obtain hydrides with different H contents and thus different Curie temperatures (TC). The phase structure and magnetocaloric effect of these hydrides were tested and analyzed. The results show that the alloy maintains the same phase structure before and after hydrogen absorption, with the main phase being a NaZn13-type cubic structure and containing a small amount of α-Fe impurity phase. As the desorption temperature increases, TC decreases approximately linearly. Due to the introduction of H atoms weakening the first-order itinerant electron metamagnetic (IEM) transition, the isothermal magnetic entropy change is reduced compared to the parent alloy. When Td $>$ 230 ℃, the magnetic entropy change decreases significantly with increasing desorption temperature, and the magnetic hysteresis decreases. When Td = 250 ℃, the magnetic entropy change curve broadens, and the first-order phase transition characteristic weakens. For the bonded sample obtained by consolidating the La0.9Ce0.1Fe11.44Si1.56Hy powder after saturated hydrogen absorption, the maximum values of adiabatic temperature change and isothermal magnetic entropy change reach 2.7 K and 7.5 J/(kg·K), respectively, under a magnetic field of 0~1.5 T.

Full Text

Magnetocaloric Effect of $La_{0}{\cdot}_{9}Ce_{0}{\cdot}_{1}Fe_{11}{\cdot}_{44}Si_{1}{\cdot}_{56}Hy$ Alloy and Powder-Bonded Bulk

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Supported by National Natural Science Foundation of China (No. 51261001) and Liaoning Provincial Natural Science Foundation (No. 2013020105)

Manuscript received: 2014-08-28; **in revised form:** 2015-01-15

Abstract

La(Fe, Si)$_{13}$-based magnetic refrigeration materials have attracted extensive attention due to their giant magnetocaloric effect (MCE), tunable Curie temperature (TC), low raw material cost, and absence of toxic elements compared to other room-temperature giant MCE materials such as $Gd_5(Ge_1\ Si\ )_4$, $MnFeP_{0 \cdot 45}As_{0 \cdot 55}$, and MnAs-based compounds. In this work, hydrogen absorption was employed to shift TC toward room temperature while maintaining a large MCE. $La_{0 \cdot 9}Ce_{0 \cdot 1}Fe_{11 \cdot 44}Si_{1 \cdot 56}Hy$ was prepared by saturated hydrogen absorption, after which the hydrogen content and TC of the hydrides were controlled by subsequent dehydrogenation at different temperatures (Td = 200–250 °C for 3 h). The phase structure and magnetocaloric effect were systematically investigated.

The results demonstrate that the samples maintain the cubic NaZn$_{13}$-type structure with a small amount of α-Fe as an impurity phase both before and after hydrogenation. TC exhibits an approximately linear decrease with increasing dehydrogenation temperature. The isothermal magnetic entropy change (ΔSm) of the hydrides decreases compared with the parent compound, primarily because the field-induced itinerant-electron metamagnetic (IEM) transition is weakened upon hydrogen absorption. For samples dehydrogenated at temperatures above 230 °C, ΔSm is remarkably reduced while magnetic hysteresis loss is simultaneously decreased. When Td = 250 °C, the ΔSm curve broadens, weakening the characteristic of the first-order phase transition. Due to the intrinsic brittleness of the hydrides, preparing samples with a specific shape is crucial for practical applications. For a magnetic field change of 1.5 T, the maximum adiabatic temperature change (ΔTad) and ΔSm for the bonded block of fully hydrogenated $La_{0 \cdot 9}Ce_{0 \cdot 1}Fe_{11 \cdot 44}Si_{1 \cdot 56}Hy$ reach approximately 2.7 K and 7.5 J/(kg•K), respectively, which are larger than those of La(Fe, Co, Si)$_{13}$ materials in the same magnetic field change range. In conclusion, bonded $La_{0 \cdot 9}Ce_{0 \cdot 1}Fe_{11 \cdot 44}Si_{1 \cdot 56}Hy$ with good MCE and different TC values have been successfully prepared and will be highly useful for practical applications in layered magnetic refrigerants at ambient temperature under low magnetic field changes in magnetic refrigerators.

KEY WORDS: $La_{0 \cdot 9}Ce_{0 \cdot 1}Fe_{11 \cdot 44}Si_{1 \cdot 56}Hy$; hydrogen absorption; dehydrogenation; powder bonding; magnetocaloric effect

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1. Introduction

Magnetic refrigeration has attracted widespread attention as a novel, highly efficient, and environmentally friendly cooling technology that is expected to replace conventional gas compression refrigeration for daily applications such as air conditioning and food preservation. This technology employs magnetic materials as refrigerants and utilizes their magnetocaloric effect (MCE). The MCE is a thermal phenomenon that occurs in magnetic ion systems during magnetization and demagnetization processes, arising from changes in magnetic entropy induced by varying external magnetic fields, accompanied by heat absorption or release. The performance of magnetocaloric materials is characterized by two key metrics: the isothermal magnetic entropy change (ΔSm) at the magnetic ordering temperature (e.g., Curie temperature TC) under different magnetic fields, and the adiabatic temperature change (ΔTad) during adiabatic magnetization at that temperature.

In 1997, Pecharsky and Gschneidner discovered giant magnetocaloric effects near room temperature in $Gd_5Si_2Ge_2$ alloy with a first-order phase transition, marking a milestone in room-temperature magnetic refrigeration research and sparking intense interest in this field. Prior to this discovery, elemental Gd (TC = 293 K) was primarily used as a room-temperature magnetic refrigerant in prototype magnetic refrigerators, but Gd exhibits a second-order phase transition with relatively small MCE. Subsequently, numerous intermetallic compounds with large MCE were discovered, including $NaZn_{13}$-type La(Fe, Si)$_{13}$-based alloys, Fe_2P-type MnFeP(As, Ge, Si) alloys, and Ni-Mn-based Heusler alloys. Among these, La(Fe, Si)$_{13}$-based alloys are particularly promising for practical applications due to their low raw material cost, non-toxicity, and MCE values up to three times that of Gd. However, their TC of approximately 190 K is far below room temperature.

To enhance the MCE of these alloys, partial substitution of La with rare earth elements (RE = Ce, Pr, Nd) has been employed to achieve giant magnetocaloric effects. To significantly increase TC, Co was added to partially replace Fe, forming La(Fe, Co, Si)$_{13}$ alloys with TC near room temperature. However, these alloys exhibit typical second-order phase transitions with substantially reduced magnetic entropy change. La(Fe, Co, Si)$_{13}$ materials have been successfully produced on a large scale and tested in magnetic refrigerators, showing relative cooling power comparable to conventional Gd. Currently, hydrogen absorption represents a highly effective method to obtain La(Fe, Si)$_{13}$-based materials with MCE far exceeding that of Gd and TC near room temperature.

Fujita and Fukamichi reported the magnetocaloric properties of La($Fe_{0}\cdot_{88}Si_{0}\cdot_{12}$)$_{13}$ and its hydride La($Fe_{0}\cdot_{88}Si_{0}\cdot_{12}$)$_{13}$Hy, showing that TC increased from 195 K for y = 0 to 330 K for y = 1.6 while the maximum magnetic entropy change and adiabatic temperature change remained nearly unchanged. The hydrogen content can be controlled by adjusting absorption temperature and pressure to obtain La(Fe, Si)$_{13}$Hy hydrides with different TC values, but this process is complex and costly. Alternatively, TC can be tuned by hydrogen absorption followed by controlled dehydrogenation. Although dehydrogenated materials are in a non-saturated hydrogenation state and may decompose when exposed to their TC environment for extended periods, potentially affecting refrigerator performance, Zimm and Jacobs demonstrated that this instability can be resolved through a thermal cycling recovery process at 60 °C in magnetic refrigeration systems, providing favorable assurance for the practical application of La(Fe, Si)$_{13}$Hy materials.

In this work, hydrides were dehydrogenated at different temperatures for a fixed duration to obtain materials with varying TC values, and the influence of dehydrogenation temperature on magnetocaloric properties was analyzed. Since hydrogenated samples become brittle and powder-like, making them unsuitable for direct use in magnetic refrigerators, magnetic refrigerants are typically fabricated as particles or flakes, such as Gd particles, Gd sheets, and La(Fe, Co, Si)$_{13}$ thin plates. In our experiments, epoxy resin powder bonding technology was employed to prepare $La_{0}\cdot_{9}Ce_{0}\cdot_{1}Fe_{11}\cdot_{44}Si_{1}\cdot_{56}$Hy bulk samples, and the magnetocaloric effect of the bonded bulk was investigated.

2. Experimental Methods

The raw materials used in this study were La and Ce with purity of 99% (mass fraction, the same below), Fe with purity of 99.9%, and Si with purity of 99.99%. Samples with the nominal composition $La_{0}\cdot_{9}Ce_{0}\cdot_{1}Fe_{11}\cdot_{44}Si_{1}\cdot_{56}$ were melted in an SK-BYL high-frequency induction furnace. The as-cast ingots were then annealed at 1200 °C for 30 h under Ar atmosphere in a high-vacuum heat treatment furnace to obtain the parent alloy. The parent alloy was crushed into uniformly sized small pieces and hydrogenated at 280 °C under a hydrogen pressure of 0.2 MPa for 5 h to achieve saturated hydride. The saturated hydride was subsequently dehydrogenated in a high-vacuum heat treatment furnace under Ar atmosphere at 200, 210, 220, 230, 240, and 250 °C for 3 h to obtain hydrides with different hydrogen contents. The saturated $La_{0}\cdot_{9}Ce_{0}\cdot_{1}Fe_{11}\cdot_{44}Si_{1}\cdot_{56}$Hy powder was lightly ground and sieved to obtain powder particles smaller than 0.2 mm. These powder particles were mixed with epoxy resin (2% mass fraction) and pressed into cylindrical bulk samples with a diameter of 12 mm and height of 16 mm under a pressure of 700 MPa. The bonded bulk samples were obtained after curing, and thin plates of various thicknesses were prepared by cutting.

Phase analysis was performed using an X' Pert Pro X-ray diffractometer (XRD). The hydrogen content was determined from thermogravimetric (TG) curves measured using a STA449C simultaneous thermal analyzer. The thermomagnetic curves (M-T, where M is magnetization and T is temperature) and isothermal magnetization curves (M-H, where H is applied magnetic field) were measured using a LakeShore 7407 vibrating sample magnetometer (VSM). The adiabatic temperature change (ΔTad) was measured using a custom-built XHY direct measurement system for magnetocaloric effects through a heating and field-decreasing process.

3. Results and Discussion

3.1 Phase Structure and Hydrogen Content

Figure 1 [Figure 1: see original paper] shows the room-temperature XRD patterns of the $La_{0}\cdot{}_{9}Ce_{0}\cdot{}_{1}Fe_{11}\cdot{}_{44}Si_{1}\cdot{}_{56}$ alloy and its hydride. The alloy maintains the same phase structure before and after hydrogen absorption, with the main phase being the cubic $NaZn_{13}$-type structure and a small amount of α-Fe impurity phase. The diffraction peaks of the hydride $La_{0}\cdot{}_{9}Ce_{0}\cdot{}_{1}Fe_{11}\cdot{}_{44}Si_{1}\cdot{}_{56}Hy$ shift significantly to lower angles compared to those before hydrogenation. This shift occurs because hydrogen atoms enter the alloy lattice as interstitial atoms, causing lattice expansion and increased unit cell volume. The lattice constant increases from 1.147 nm to 1.158 nm. The diffraction peaks of the α-Fe phase remain unchanged after hydrogenation, indicating that the α-Fe impurity phase does not absorb hydrogen and that the $NaZn_{13}$ main phase is responsible for hydrogen absorption. The introduction of interstitial hydrogen atoms increases the distance between magnetic Fe atoms and enhances interatomic exchange interactions, leading to increased TC and modified magnetic properties.

To determine the hydrogen content of the hydride $La_{0}\cdot{}_{9}Ce_{0}\cdot{}_{1}Fe_{11}\cdot{}_{44}Si_{1}\cdot{}_{56}Hy$ after hydrogen absorption, TG curves were measured, as shown in Figure 2 [Figure 2: see original paper]. The hydride exhibits continuous weight loss between 200 and 420 °C, with a weight loss fraction of approximately 0.19%, from which the hydrogen content was estimated to be y = 1.56.

3.2 Magnetic Properties and Magnetocaloric Effect

Figure 3 [Figure 3: see original paper] presents the thermomagnetic curves and isothermal magnetic entropy change curves calculated from isothermal magnetization curves near TC using the Maxwell relation for the $La_{0}\cdot{}_{9}Ce_{0}\cdot{}_{1}Fe_{11}\cdot{}_{44}Si_{1}\cdot{}_{56}$ alloy before and after hydrogen absorption. TC can be determined from the M-T curves by finding the temperature at which dM/dT reaches its minimum value. At TC, a sharp change in magnetic moment occurs, corresponding to the transition from ferromagnetism to paramagnetism, indicating a large magnetic entropy change. For the $La_{0}\cdot{}_{9}Ce_{0}\cdot{}_{1}Fe_{11}\cdot{}_{44}Si_{1}\cdot{}_{56}$ alloy, TC increases from 195 K before hydrogen absorption to 315 K after absorption, a rise of 120 K that brings it near room temperature. The magnetic entropy change decreases from 13.75 J/(kg · K) to 10.35 J/(kg · K) because hydrogen absorption weakens the first-order itinerant-electron metamagnetic (IEM) transition characteristics. Although the magnetic entropy change is reduced, it remains at a large value. The introduced hydrogen atoms do not alter the IEM transition behavior, which is related to the band structure of Fe 3d electrons. Interstitial hydrogen atoms have minimal influence on the density of states near the Fermi surface of Fe 3d electrons, thus preserving the large magnetic entropy change.

To obtain hydrides with different TC values near room temperature, the saturated hydride was annealed at various temperatures. Since H_2 becomes unstable above 150 °C and desorbs from the alloy, reducing hydrogen content, dehydrogenation can be used to control TC. Figure 4a [Figure 4: see original paper] shows the thermomagnetic curves of the saturated hydride $La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}$Hy after 3 h dehydrogenation at 200, 210, 220, 230, 240, and 250 °C under an applied magnetic field of 0.05 T. The relationship between TC and dehydrogenation temperature Td (Figure 4b) reveals that the TC values of the dehydrogenated hydrides are 303, 294, 289, 285, 281, and 271 K, respectively. The saturated hydride has a TC of 315 K, which decreases to 303 K after dehydrogenation at 200 °C for 3 h. When the dehydrogenation temperature increases to 250 °C, TC drops below 0 °C. With constant dehydrogenation time, increasing the dehydrogenation temperature leads to a continuous decrease in TC because hydrogen atoms, present as interstitials in the alloy lattice, gradually diffuse out at higher temperatures. Notably, TC decreases approximately linearly with increasing dehydrogenation temperature.

Figures 5a [Figure 5: see original paper] and 6a [Figure 6: see original paper] show the isothermal magnetization curves measured near their respective TC values for $La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}$Hy dehydrogenated at 200 and 250 °C for 3 h. The measurement temperature interval was 2 K, and the applied magnetic field varied from 0 to 1.5 T. Figures 5b and 6b present the corresponding Arrott plots. The magnetization curves reveal that no field-induced magnetic moment jump occurs even at the maximum magnetic field of 1.5 T, indicating the absence of a clear itinerant-electron metamagnetic transition. As the dehydrogenation temperature increases, the change in magnetization with magnetic field becomes progressively slower, suggesting that the field-induced first-order phase transition characteristic is weakened. Concurrently, magnetic hysteresis decreases significantly, with the maximum hysteresis reducing from 3.55 J/kg for the sample dehydrogenated at 200 °C to 1.68 J/kg for the sample dehydrogenated at 250 °C.

According to the itinerant-electron s-d band model, the slope of Arrott plots can be used to characterize the type of magnetic phase transition. A positive slope in Arrott curves slightly above TC indicates a second-order phase transition, whereas a negative slope or inflection point signifies a first-order phase transition. The Arrott curves for the hydride dehydrogenated at 200 °C show inflection points, confirming a field-induced first-order phase transition. In contrast, the Arrott curves for the hydride dehydrogenated at 250 °C exhibit no clear inflection points, indicating that the first-order phase transition characteristic gradually weakens with increasing dehydrogenation temperature. The powder-like nature of the hydrogenated alloy increases the specific surface area and reduces internal stress, thereby decreasing hysteresis loss. Similar to the effect of B addition in La(Fe, Si)$_{13}$-based alloys, where B enters the parent alloy as interstitial atoms and reduces both ΔSm and magnetic hysteresis while weakening the field-induced first-order transition, temperature has a more pronounced effect on hydride properties. As dehydrogenation temperature increases, the first-order transition characteristic weakens, likely due to instability of the hydride at elevated temperatures, which may cause significant lattice structural changes and substantially alter magnetocaloric performance.

Based on the Maxwell relation, the isothermal magnetic entropy change under a magnetic field variation of 0–1.5 T was calculated from the isothermal magnetization curves for hydrides dehydrogenated at different temperatures, as shown in Figure 7 [Figure 7: see original paper]. The maximum isothermal magnetic entropy changes (-ΔS) are 10.81, 9.84, 9.28, 9.99, 8.74, and 7.19 J/(kg·K) for dehydrogenation temperatures of 200, 210, 220, 230, 240, and 250 °C, respectively. For Td $\leq$ 230 °C, the dehydrogenation temperature has a minor effect on the isothermal magnetic entropy change, with -ΔS remaining around 10 J/(kg·K). However, when Td > 230 °C, the isothermal magnetic entropy change decreases significantly with increasing dehydrogenation temperature. At Td = 250 °C, the magnetic entropy change value is lower and the full width at half maximum broadens, indicating weakened first-order phase transition characteristics. Compared with the parent alloy $La_{0\cdot 9}Ce_{0\cdot 1}Fe_{11\cdot 44}Si_{1\cdot 56}$ (-ΔS = 13.75 J/(kg·K)), all hydrides show reduced isothermal magnetic entropy change values due to weakened first-order IEM transition upon hydrogen absorption. While adjusting the dehydrogenation temperature at constant time effectively tunes the TC of hydrides, it is important to note that temperature significantly affects dehydrogenation behavior, and excessively high dehydrogenation temperatures lead to substantially reduced isothermal magnetic entropy change. Therefore, the dehydrogenation temperature must be appropriately controlled.

3.3 Magnetocaloric Properties of Bonded Bulk

Cylindrical bulk samples of $La_{0\cdot 9}Ce_{0\cdot 1}Fe_{11\cdot 44}Si_{1\cdot 56}$Hy were prepared through powder bonding after grinding the saturated hydride. The adiabatic temperature change and isothermal magnetic entropy change curves under a varying magnetic field of 0–1.5 T were obtained through direct measurement and M-H curve analysis, as shown in Figure 8 [Figure 8: see original paper]. The bonded bulk exhibits a large magnetocaloric effect near room temperature, with ΔTad = 2.7 K and ΔSm = 7.5 J/(kg·K) at TC = 307 K. The TC of the bonded bulk (307 K) is slightly lower than that of the powder before bonding (315 K), likely because heating during the curing process caused partial hydrogen desorption from the bulk. Although the magnetocaloric performance of the bonded magnet is reduced due to powdering and the addition of a small amount of nonmagnetic epoxy resin, it remains superior to that of La(Fe, Co, Si)$_{13}$ materials measured under the same magnetic field. For example, $LaFe_{11}Co_{0\cdot 9}Si_{1\cdot 1}$ shows

ΔTad = 1.5 K at TC = 295 K, while $\mathrm{LaFe_{11}Co_{0\cdot9}Si_{1\cdot1}B_{0\cdot25}}$ exhibits ΔTad = 2.3 K and ΔSm = 5.2 J/(kg·K) at TC = 291 K. Therefore, bulk hydride refrigerant materials with different Curie temperatures can be obtained through bonding, enabling the preparation of layered composite magnetic refrigerants for magnetic refrigerators.

4. Conclusions

1. The $\mathrm{La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}}$ alloy retains the $\mathrm{NaZn_{13}}$-type cubic structure after hydrogen absorption, with TC increasing by 120 K to near room temperature. The introduction of interstitial hydrogen atoms into the alloy lattice increases the lattice constant and weakens the first-order itinerant-electron metamagnetic transition characteristic, resulting in reduced isothermal magnetic entropy change values.

2. Dehydrogenation for 3 h at temperatures of 200–250 °C causes TC to decrease approximately linearly with increasing Td, dropping below 0 °C at Td = 250 °C. For Td $\leq$ 230 °C, the dehydrogenation temperature has a minor effect on the isothermal magnetic entropy change, with the maximum value (-ΔS) remaining around 10 J/(kg·K) under a magnetic field variation of 0-1.5 T. When Td > 230 °C, -ΔS decreases significantly with increasing dehydrogenation temperature, reaching 7.19 J/(kg·K) at Td = 250 °C, with broadened full width at half maximum and weakened first-order phase transition characteristics. Since excessively high dehydrogenation temperatures substantially reduce the isothermal magnetic entropy change, the dehydrogenation temperature must be appropriately controlled.

3. The saturated $\mathrm{La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}Hy}$ powder was ground and bonded into cylindrical bulk samples, achieving superior magnetocaloric performance compared to La(Fe, Co, Si)$_{13}$. Under an applied magnetic field of 0–1.5 T, the maximum adiabatic temperature change and isothermal magnetic entropy change reach 2.7 K and 7.5 J/(kg·K), respectively.

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