Magnetocaloric Effect of La_{0.9}Ce_{0.1}Fe_{11.44}Si_{1.56}H_ Alloy and Its Powder-Bonded Bulk Postprint

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Date: 2016-11-10T00:00:00+00:00

Abstract

La0.9Ce0.1Fe11.44Si1.56 alloy was subjected to saturated hydrogen absorption, followed by dehydrogenation treatment at different temperatures (Td = 200–250 °C) for 3 h to obtain hydrides with varying hydrogen contents and thus different Curie temperatures (TC). The phase structure and magnetocaloric effect were characterized and analyzed. The results indicate that the alloy maintains the same phase structure before and after hydrogen absorption, with the primary phase being NaZn13-type cubic structure and containing a minor amount of α-Fe impurity phase. As the dehydrogenation temperature increases, TC decreases approximately linearly. The introduction of H atoms weakens the first-order itinerant electron metamagnetic (IEM) transition, resulting in a reduced isothermal magnetic entropy change compared to the parent alloy. When Td > 230 °C, the magnetic entropy change decreases significantly with increasing dehydrogenation temperature, and the magnetic hysteresis diminishes. At Td = 250 °C, the magnetic entropy change curve broadens, and the first-order phase transition characteristics are weakened. For the bonded sample obtained by solidifying saturated hydrogenated La0.9Ce0.1Fe11.44Si1.56Hy powder, the maximum adiabatic temperature change and isothermal magnetic entropy change reach 2.7 K and 7.5 J/(kg?K), respectively, under a magnetic field variation 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 BLOCK

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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

Recently, $La(Fe,Si)_{13}$-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 $Gd_5(Ge_1\ Si\ )_4$, $MnFeP_{0\cdot45}As_{0\cdot55}$, and MnAs-based compounds. In this work, hydrogen absorption was employed to shift the TC to near room temperature while maintaining a large MCE. $La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}$ hydride 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 investigated systematically.

The results demonstrate that all samples possess the cubic $NaZn_{13}$-type structure with a small amount of α-Fe impurity phase. The TC exhibits an approximately linear decrease with increasing dehydrogenation temperature. The isothermal magnetic entropy change (ΔSm) of the hydrides decreases compared with the parent alloy, which is mainly attributed to the weakening of the field-induced itinerant-electron metamagnetic (IEM) transition upon hydrogen absorption. For samples desorbed at temperatures above 230 °C, ΔSm decreases remarkably while the magnetic hysteresis loss is favorably reduced simultaneously. With further increasing temperature to 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 specific shapes 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 $La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}$ hydride are 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 $La_{0\cdot9}Ce_{0\cdot1}Fe_{11\cdot44}Si_{1\cdot56}$ hydrides with good MCE and different TC values have been successfully prepared and will be very useful for practical applications as layered magnetic refrigerants at ambient temperature under low 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, where changes in the external magnetic field induce changes in the magnetic entropy of the material, 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) 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 the first-order phase transition material $Gd_5Si_2Ge_2$, marking a significant 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 undergoes a second-order phase transition with relatively small MCE. Subsequently, numerous intermetallic compounds with large MCE have been identified, 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, partial substitution of La with rare earth elements (RE = Ce, Pr, Nd) has been employed, yielding giant magnetocaloric effects. To significantly increase TC, Co has been added to replace part of 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. Although $La(Fe,Co,Si)_{13}$ materials have been successfully scaled up for magnetic refrigerator testing, their relative cooling capacity is only comparable to conventional Gd.

Currently, hydrogen absorption has proven to be a highly effective method for obtaining $La(Fe,Si)_{13}$-based materials with TC near room temperature while maintaining MCE values far exceeding that of Gd. Fujita and Fukamichi reported that for $La(Fe_{0 \cdot 88}Si_{0 \cdot 12})_{13}$ and its hydride $La(Fe_{0 \cdot 88}Si_{0 \cdot 12})_{13}Hy$, 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. However, optimizing the hydrogen absorption process is complex and costly. Therefore, a post-absorption dehydrogenation method can be used to tune TC. Although hydrogen may decompose and become unstable when exposed to temperatures near TC 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 a favorable guarantee for the application of $La(Fe,Si)_{13}Hy$ materials.

In this work, saturated hydrides were subjected to dehydrogenation at different temperatures for a fixed duration to obtain hydrides with various 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 this study, 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 blocks was investigated.

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2. EXPERIMENTAL

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%. Starting materials 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 alloy was crushed into small pieces of uniform size and subjected to hydrogen absorption at 280 °C under a hydrogen pressure of 0.2 MPa for 5 h to obtain saturated hydride. The saturated hydride was then placed in a high-vacuum heat treatment furnace and annealed under Ar protection for 3 h at 200, 210, 220, 230, 240, and 250 °C to perform dehydrogenation experiments, yielding 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 blocks with a diameter of 12 mm and height of 16 mm under a pressure of 700 MPa. After curing, bonded blocks were obtained, and thin plates of various thicknesses were cut from these blocks for further characterization.

Phase analysis was performed using an X' Pert Pro X-ray diffractometer (XRD). The hydrogen content was determined from thermogravimetric (TG) curves measured with a STA449C simultaneous thermal analyzer. Magnetization-temperature (M-T) curves and isothermal magnetization curves (M-H) 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, employing a heating and field-decreasing measurement protocol.

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3. RESULTS AND DISCUSSION

3.1 Phase Structure and Hydrogen Content [Figure 1: see original paper] shows the room-temperature XRD patterns of the $La_0{\cdot}_9Ce_0{\cdot}_1Fe_{11}{\cdot}_{44}Si_1{\cdot}_{56}$ alloy and its hydride. The alloy maintains the same phase structure before and after hydrogen absorption, with the main phase being $NaZn_{13}$-type cubic structure accompanied by a small amount of α-Fe impurity phase. The diffraction peaks of the hydride $La_0{\cdot}_9Ce_0{\cdot}_1Fe_{11}{\cdot}_{44}Si_1{\cdot}_{56}$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 α-Fe phase diffraction peaks 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 incorporation of interstitial hydrogen atoms increases the distance between magnetic Fe atoms and enhances their exchange interaction, leading to increased TC and modified magnetic properties.

To determine the hydrogen content in the hydride $La_0{\cdot}_9Ce_0{\cdot}_1Fe_{11}{\cdot}_{44}Si_1{\cdot}_{56}$Hy, TG curves were measured, as shown in [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: see original paper] presents the magnetization-temperature (M-T) curves and isothermal magnetic entropy change curves calculated from isothermal magnetization curves near TC using the Maxwell relation for the $La_0{\cdot}_9Ce_0{\cdot}_1Fe_{11}{\cdot}_{44}Si_1{\cdot}_{56}$ alloy before and after hydrogen absorption. The Curie temperature can be determined from the M-T curves by finding the temperature at which dM/dT reaches its minimum value. At TC, a sharp magnetic moment transition occurs, corresponding to the ferromagnetic-to-paramagnetic transition and indicating a large magnetic entropy change. For the $La_0{\cdot}_9Ce_0{\cdot}_1Fe_{11}{\cdot}_{44}Si_1{\cdot}_{56}$ alloy, TC increases from 195 K before hydrogen absorption to 315 K after absorption, a remarkable increase of 120 K that brings it to near room temperature. The magnetic entropy change decreases from 13.75 J/(kg • K) to 10.35 J/(kg • K). This reduction is attributed to hydrogen absorption weakening the first-order itinerant electron metamagnetic (IEM) transition characteristics. Although the magnetic entropy change decreases, it remains at a large value. The incorporated 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 subjected to annealing at various temperatures. Since H_2 becomes unstable above 150 °C and desorbs from the alloy, leading to reduced hydrogen content, dehydrogenation can be employed to control TC. [Figure 4a: see original paper] shows the M-T curves of the saturated hydride $La_{0}{\cdot}_{9}Ce_{0}{\cdot}_{1}Fe_{11}{\cdot}_{44}Si_{1}{\cdot}_{56}$Hy after 3 h dehydrogenation at 200, 210, 220, 230, 240, and 250 °C under an applied field of 0.05 T. The relationship between TC and dehydrogenation temperature Td is shown in [Figure 4b: see original paper]. After dehydrogenation, the TC values 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 continuously reduces TC because hydrogen atoms exist as interstitials in the alloy lattice and gradually diffuse out at higher temperatures. The data clearly show that TC decreases approximately linearly with increasing dehydrogenation temperature.

3.3 Effect of Dehydrogenation Temperature on Phase Transition Characteristics [Figure 5a: see original paper] and [Figure 6a: see original paper] show the isothermal magnetization curves measured near their respective TC values for $La_{0}{\cdot}_{9}Ce_{0}{\cdot}_{1}Fe_{11}{\cdot}_{44}Si_{1}{\cdot}_{56}$Hy after 3 h dehydrogenation at 200 and 250 °C. The measurements were performed at temperature intervals of 2 K under an applied field of 0–1.5 T. [Figure 5b: see original paper] and [Figure 6b: see original paper] display the corresponding Arrott plots. The magnetization curves reveal that no field-induced magnetic moment transition occurs even at the maximum field of 1.5 T, indicating the absence of obvious itinerant electron metamagnetic transition. Moreover, as the dehydrogenation temperature increases, the magnetic moment changes more slowly with the magnetic field, suggesting that the field-induced first-order transition characteristic weakens while magnetic hysteresis decreases significantly. The maximum hysteresis decreases from 3.55 J/kg for the sample dehydrogenated at 200 °C to 1.68 J/kg for the sample treated at 250 °C.

According to the itinerant electron s-d orbital model, the slope of Arrott plots can be used to characterize the nature of magnetic phase transitions. A positive slope of Arrott curves slightly above TC indicates a second-order phase transition, whereas a negative slope or inflection point suggests a first-order transition. The Arrott plots for the hydride dehydrogenated at 200 °C show inflection points, confirming a field-induced first-order transition. In contrast, the Arrott plots for the 250 °C dehydrogenated sample show no obvious inflection points, indicating that the first-order transition characteristic gradually weakens with increasing dehydrogenation temperature. The powder formation after hydrogen absorption increases the specific surface area and reduces internal stress, thereby decreasing hysteresis loss. Similar effects have been observed in $La(Fe,Si)_{13}$-based alloys doped with B, where B also enters the lattice as interstitial atoms, causing gradual decreases in isothermal magnetic entropy change and hysteresis while weakening the field-induced first-order transition characteristic as B content increases. Temperature has a more pronounced effect on hydride properties, with increasing dehydrogenation temperature gradually weakening the first-order transition characteristic, likely related to hydride instability at elevated temperatures, which may induce significant lattice structural changes and substantially alter magnetocaloric performance.

3.4 Magnetic Entropy Change of Hydrides Based on the Maxwell relation, the isothermal magnetic entropy change was calculated from the isothermal magnetization curves under a magnetic field change of 0–1.5 T for hydrides dehydrogenated at various temperatures, as shown in [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 minimal 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 relatively low with broadened half-width, indicating weakened first-order 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 exhibit reduced isothermal magnetic entropy change due to hydrogen absorption weakening the first-order IEM transition. While adjusting the dehydrogenation temperature at constant time can effectively tune the TC of hydrides, it is important to note that temperature significantly affects dehydrogenation behavior, and excessively high dehydrogenation temperatures lead to substantial reduction in isothermal magnetic entropy change. Therefore, proper control of dehydrogenation temperature is essential.

3.5 Magnetocaloric Properties of Bonded Samples 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 magnetic field change of 0–1.5 T are shown in [Figure 8: see original paper]. The bonded samples exhibit large magnetocaloric effects near room temperature, with $\Delta Tad = 2.7$ K and $\Delta Sm = 7.5$ J/(kg · K) at TC = 307 K. The TC of the bonded sample (307 K) is slightly lower than that before bonding (315 K), likely due to partial hydrogen desorption during the curing process when the bonded block was heated.

Although the magnetocaloric performance of the bonded magnet decreases due to powdering during preparation and the addition of a small amount of non-magnetic epoxy resin, the values remain larger than those of $\mathrm{La(Fe,Co,Si)}_{13}$ materials measured under the same magnetic field. For example, $\mathrm{LaFe}_{11}\mathrm{Co}_{0\cdot 9}\mathrm{Si}_{1\cdot 1}$ exhibits $\Delta\mathrm{Tad} = 1.5$ K at TC = 295 K, while $\mathrm{LaFe}_{11}\mathrm{Co}_{0\cdot 9}\mathrm{Si}_{1\cdot 1}\mathrm{B}_{0\cdot 25}$ shows $\Delta\mathrm{Tad} = 2.3$ K and $\Delta\mathrm{Sm} = 5.2$ J/(kg • K) at TC = 291 K. Therefore, bulk hydride refrigerant materials with different Curie temperatures can be obtained through bonding methods for fabrication of multilayer composite magnetic refrigerants in magnetic refrigerators.

4. CONCLUSIONS

1. The $\mathrm{La}_{0\cdot 9}\mathrm{Ce}_{0\cdot 1}\mathrm{Fe}_{11\cdot 44}\mathrm{Si}_{1\cdot 56}$ alloy maintains the NaZn_{13}-type cubic structure after hydrogen absorption, with TC increasing by 120 K to near room temperature. The incorporation of interstitial hydrogen atoms expands the lattice constant and weakens the first-order itinerant electron metamagnetic transition characteristic, resulting in reduced isothermal magnetic entropy change values.

2. Dehydrogenation at temperatures of 200–250 °C for 3 h 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 minimal effect on the isothermal magnetic entropy change, with the maximum value ($-\Delta\mathrm{S}$) remaining around 10 J/(kg • K) under a 0–1.5 T field change. When Td > 230 °C, $-\Delta\mathrm{S}$ decreases significantly with increasing dehydrogenation temperature, reaching 7.19 J/(kg • K) at Td = 250 °C with broadened half-width and weakened first-order transition characteristics. Since excessively high dehydrogenation temperatures substantially reduce the isothermal magnetic entropy change of hydrides, proper temperature control is necessary.

3. The saturated $\mathrm{La}_{0\cdot 9}\mathrm{Ce}_{0\cdot 1}\mathrm{Fe}_{11\cdot 44}\mathrm{Si}_{1\cdot 56}\mathrm{Hy}$ powder was successfully bonded into cylindrical bulk samples with superior magnetocaloric performance compared to $\mathrm{La(Fe,Co,Si)}_{13}$ materials. Under an applied 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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