Influence of High-Pressure Nitrogenation on the Structural, Magnetic and Magnetocaloric Properties of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$

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Received: 21 August 2015 / Revised: 20 October 2015 / Published online: 13 November 2015
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Abstract Nitrogenation of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ with a particle size of 100–150 μm was performed in a high-purity N_2 atmosphere of 40 MPa. $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$ nitrogenated at 480 °C exhibits an increase in the Curie temperature T_C from 187 to 195 K. Moreover, for a field change from 0 to 1.5 T, the maximum hysteresis loss at T_C is remarkably reduced from 53 to 3 J kg^{-1} and a large magnetic-entropy change ΔS_m of over 10 $\text{J kg}^{-1} \text{K}^{-1}$ is maintained. For a $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ sample, nitrogenated at 550 °C, the XRD pattern clearly exhibits two 1:13 phases and, accordingly, the thermal magnetic curves exhibit two transitions, at 210 and at 295 K. The two transitions lead, for a field change of 5 T, to a ΔS_m larger than 2 $\text{J kg}^{-1} \text{K}^{-1}$ over a large temperature range from 200 to 310 K with a maximum value of 5.3 $\text{J kg}^{-1} \text{K}^{-1}$ at 225 K. Upon further increase in the nitrogenation temperature to 650 °C, the amount of the nitrogen-poor phase strongly decreases and a large amount of $\alpha\text{-Fe}$ precipitates, resulting in a large reduction in the MCE.

KEY WORDS: Metals and alloys; Magnetocaloric effect; Magnetic hysteresis

1 Introduction

Nowadays, increasing interest is paid to $\text{La}(\text{Fe}_{1-x}\text{Si}_x)_{13}$ compounds due to their low material costs, non-toxic constituting elements and large magnetocaloric effect (MCE) compared to other room-temperature giant MCE materials such as $\text{Gd}_5(\text{Ge}_{1-x}\text{Si}_x)_4$ [1], $\text{MnFeP}_{0.45}\text{As}_{0.55}$ [2] and MnAs -based compounds [3, 4]. The large MCE of $\text{La}(\text{Fe}_{1-x}\text{Si}_x)_{13}$ compounds is associated with a temperature-induced paramagnetic–ferromagnetic (PM–FM) transition and with a field-induced itinerant-electron

metamagnetic (IEM) transition [5]. Both transitions are accompanied by a large volume increase in up to 1.35% [6], which causes large thermal and magnetic hysteresis and confines the practical use of the materials. Furthermore, the Curie temperature T_C of the $\text{La}(\text{Fe}_{1-x}\text{Si}_x)_{13}$ compounds is about 200 K. As a consequence, much effort has been made to reduce the hysteresis and to increase T_C . In the $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.5}\text{Si}_{1.5}\text{C}_{0.3}$ compound, T_C has increased to 211 K, and meanwhile, a large MCE is maintained [7]. However, a large hysteresis loss of 23 $\text{J kg}^{-1} \text{K}^{-1}$ still remains. The introduction of interstitial hydrogen atoms increases T_C to room temperature without seriously reducing the MCE. However, the chemical stability of the hydrides is a problem. Addition of boron has increased the T_C of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ from 183 to 230 K, but unfortunately, the MCE is largely reduced because the nature of the magnetic transition has been changed from first order to second order [8].

The intrinsic magnetic properties, especially the magnetic anisotropy, of many rare earth transition metal compounds, such as R_2Fe_{17} and RFe_{11}Ti (R is the rare earth

Available online at <http://link.springer.com/journal/40195>

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element), have been found to be improved dramatically due to the introduction of interstitial nitrogen [9, 10]. Nitrogenation also has strong effects on the cubic LaZn_{13} -type $\text{La}(\text{Fe}, \text{Co})_{13}$ -based compounds, including a large lattice expansion and increase in T_C and the spontaneous magnetization [11–13]. So far, only a few studies have been focused on the influence of interstitial nitrogen on the MCE of $\text{La}(\text{Fe}_{1-x}\text{Si}_x)_{13}$ -based compounds. Insertion of 1.3 nitrogen atoms per $\text{LaFe}_{11.7}\text{Si}_{1.3}$ formula increases the lattice constant from 11.467 to 11.733 Å and the T_C from 190 to 230 K. However, the MCE is greatly reduced from 28 to 3.5 J kg⁻¹ K⁻¹ when the field changes from 0 to 5 T because the nature of the magnetic phase transition has changed to second order [14]. In general, nitrogenation is carried out at low N_2 pressure and, in order to make the gas–solid reaction proceed more completely, the material is crushed into the particles smaller than 45 µm for increasing the sample surface. However, when bulk material is broken into small particles, the MCE can be greatly reduced [15, 16]. Higher N_2 pressure is expected to increase the homogenization of nitrogenation. In the present work, nitrogenation of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ with a particle size of 100–150 µm has been performed in a high-purity N_2 atmosphere of 40 MPa. The influence of high-pressure nitrogenation at different temperatures on the structure, magnetic and MCE properties of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ has been systematically investigated.

2 Experimental

The parent compound $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ was synthesized in an induction furnace. The starting materials are Pr, La, Fe and Fe with commercial purity of 99, 99, 99 and 99.99%, respectively, which were mixed and the obtained ingots were annealed at 1050 °C for 10 days. The samples were then ground to particles of 100–150 µm and nitrogenated by using a self-made furnace in a high-purity N_2 atmosphere of 40 MPa at 480, 550 and 650 °C. The N contents in the $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds were determined from the weight difference before and after nitrogenation to be $x = 0.3, 1.2$ and 2.1, respectively.

X-ray diffraction (XRD) was performed using CuK_α radiation with a Rigaku *d*/Max- γ A diffractometer. The magnetic properties were measured using a superconducting quantum interference device (SQUID) magnetometer and a vibrating-sample magnetometer (Lakeshore 7407) (100 K $\leq T \leq$ 400 K, 0 T $\leq B \leq$ 5 T). The magnetic-entropy change ΔS_m was derived from M–B curves, measured at various temperatures close to the phase transition.

3 Results and Discussion

Figure 1 shows the XRD patterns of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ ($x = 0, 0.3, 1.2, 2.1$). The results indicate that the prepared $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ parent bulk compound possesses a cubic NaZn_{13} -type structure with a small amount of α -Fe as impurity phase. For $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$ nitrogenated at 480 °C, the (531), (422) and (420) reflections of the NaZn_{13} -type main phase begin to separate and, for $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ nitrogenated at 550 °C, the XRD pattern clearly exhibits two 1:13 phases, indicating a two-phase mixture of nitrogen-poor and nitrogen-rich phases. It can be clearly seen that the reflections of the cubic NaZn_{13} -type structure for the nitrogen-rich phase shift to lower angles, indicating larger lattice parameters and higher contents of N. In accordance with the two phases with different N contents, the thermal magnetization curves of the $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ sample exhibit two transitions at 210 and at 295 K (as shown in Fig. 3a). The inhomogeneity of nitrogenation for the two samples can be ascribed to the comparative low nitrogenation temperature and the large nitrogenated particle size of 100–150 µm in the present nitrogenation study. It has been previously reported that the minimum nitrogenation temperature for $\text{LaFe}_{13-x}\text{Si}_x$ compounds is 650 °C under an ambient pressure [13]. The present study suggests that, despite the high pressure of 40 MPa, the nitrogenation temperature of 550 °C is still too low to obtain complete and homogeneous nitrogenation. In previous experiments, the

Fig. 1 XRD patterns of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ ($x = 0, 0.3, 1.2$ and 2.1) compounds nitrogenated at different temperatures

nitrogenated particles were smaller than 45 μm [13] and it is understandable that, at larger particle size, it is more difficult to achieve a homogeneous distribution of the nitrogen atoms in the material. With further increase in the nitrogenation temperature to 650 $^{\circ}\text{C}$, the amount of the nitrogen-rich phase further increases, while the amount of the nitrogen-poor phase strongly decreases. It is worthwhile noting that a large amount of $\alpha\text{-Fe}$ precipitates. In the previous work [17], a similar precipitation of large amounts of Mo and $\alpha\text{-Fe}$ from the main phase has been observed in $\text{SmFe}_{10}\text{Mo}_2$ with increase in the nitrogenation pressure to 40 MPa.

The temperature dependence of the magnetization of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds with $x = 0.0$ and 0.3 was measured in a field of 0.05 T during the warming process. For comparison, the parent compound $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ with the same particle size of 100–150 nm was measured. As shown in Fig. 2, with increasing temperature, a first-order magnetic phase transition from the FM to PM state takes place at T_C of 186 K and 195 K for $x = 0.0$ and 0.3 , respectively. T_C has been defined as the temperature corresponding to the minimum in the temperature dependence of the first derivative of the magnetization (dM/dT). Previously, it has been observed that the introductions of the interstitial hydrogen, boron or carbon atoms also cause the Curie temperature to increase due to the variation of Fe–Fe bond length and both the lattice parameter a and T_C are found to increase linearly with the interstitial-atom contents [13, 18]. It has been reported that the Fe–Fe interaction is a key factor that influences the magnetic properties of Fe-rich rare earth iron compounds and the Curie temperature strongly depends on the Fe–Fe distance [19]. Neutron diffraction has shown that the N atoms in $\text{LaFe}_{11}\text{Al}_2$ nitrides occupy exclusively the 24d interstitial site [20]. Thus, the increase in T_C in the present

$\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ can be attributed to variation of the Fe–Fe bond length caused by the interstitial nitrogen atoms.

The temperature dependences of the magnetization of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ and $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{2.1}$, measured in a zero-field-cooled process (ZFC) and field-cooled process (FC), are shown in Fig. 3. The magnetization of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ begins to decrease gradually above 200 K, and there are two minima in the dM/dT curves, suggesting that, in accordance with the two 1:13 phases with different nitrogen contents, two transitions occur at about 210 and 295 K (as shown in the inset of Fig. 3a). This result is in accordance with the XRD results in Fig. 1. A previous study [13] indicated that T_C of $\text{La}(\text{Fe},\text{Si})_{13}$ compounds can be increased to the room temperature upon nitrogenation. The thermal magnetic curves of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{2.1}$ show almost the same behavior as $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ with two magnetic transitions at 200 and 290 K (as shown in the inset of Fig. 3b). The lower T_C can be ascribed to the larger amount of precipitation of $\alpha\text{-Fe}$, which leads to a change of the composition of the main phase. It has been reported that T_C of $\text{La}(\text{Fe},\text{Si})_{13}$ hydrides is very sensitive to the hydrogen content [21], and thus, inhomogeneous distribution of the nitrogen atoms can lead to the quite different transition temperatures of the present $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ nitrides.

To explore the effect of high-pressure nitrogenation on the field-induced IEM transition and especially on the hysteresis loss, the magnetic field dependence of the magnetization ($M(B)$) was measured. As shown in Fig. 4a, b, at temperatures close to T_C , after an initial linear increase at low fields, which reveals that the compound is in the paramagnetic state, the magnetization of

Fig. 2 Temperature dependence of the magnetization at 0.05 T of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds with $x = 0$ and 0.3

Fig. 3 Temperature dependence of the magnetization at 0.05 T of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds with $x = 1.2$ **a** and 2.1 **b**, where the insets show the first derivative of the magnetization (dM/dT) as a function of temperature

Fig. 4 Field dependence of the magnetization of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds at temperatures near T_C for $x = 0$ **a** and $x = 0.3$ **b**

$\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ shows an abrupt increase, indicating a field-induced first-order IEM transition. In sharp contrast with this, however for $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$, no clear inflection point is observed in the $M(B)$ curves showing the weakness of the first-order transition. It is clearly seen that the magnetic hysteresis, defined as the enclosed area in the field cycle shown in Fig. 4, remarkably decreases upon the introduction of nitrogen. The maximum hysteresis loss is 53 J kg^{-1} for the bulk parent compound and 3 J kg^{-1} for $x = 0.3$, showing an enormous decrease in the hysteresis loss after high-pressure nitrogenation. When the average particle size of the fragments is below $100 \mu\text{m}$, it exhibits a lower operating field for the magnetic transition and smaller hysteresis loss due to the reduction in internal strain [15, 16]. Also, some extrinsic factors, such as sample geometry and the conditions for heat transfer, can influence the magnetic hysteresis [22, 23]. In addition, during the first-order structural transition, the internal strain can influence the hysteresis by confining the volume expansion [6, 24]. Recently, it has been reported that in plate-shaped $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ hydrides sintered in high-pressure H_2 atmosphere, hysteresis is almost absent due to the existence of the micropores and a large size distribution ranging from several micrometers to about $100 \mu\text{m}$ [25]. As stated above, the sizes of both the parent alloy and the nitride particles are $100\text{--}150 \mu\text{m}$, and the physical mechanism of the hysteresis reduction in $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ nitrides can be ascribed to some intrinsic factors. The influence of hydrogenation on the magnetism of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ has been interpreted by the renormalization effect of spin fluctuations based on the Landau–Ginzburg theory [19]. Similarly, in the present $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds, the interstitial nitrogen may modify the spin fluctuations at T_C and lower the energy barrier separating the PM and FM state, leading to considerable improvement of the magnetic hysteresis behavior.

Fig. 5 Field dependence of the magnetization of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds at temperatures near T_C for $x = 1.2$ **a** and $x = 2.1$ **b**

As shown in Fig. 5a, b, the magnetic isotherms of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ and $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{2.1}$ show second-order-transition characteristics. The magnetic field-induced IEM transition near the Curie temperature is not observed. These results indicate that the first-order transition has become much weaker after nitrogenation.

The magnetic-entropy change $\Delta S_m(T, B)$ for a magnetic field change is given by the Maxwell relationship: $\Delta S_m(T, B) = S(T, B) - S(T, 0) = \int_0^B \left(\frac{\partial M}{\partial T} \right)_B dB$. Figure 6a displays the temperature dependence of ΔS_m of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ and $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$ for a magnetic field change from 0 to 1.5 T. The maximal ΔS_m for the parent compound $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ with particle size $100\text{--}150 \mu\text{m}$ is $23 \text{ J kg}^{-1} \text{ K}^{-1}$ at 184 K. The maximal ΔS_m of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$ has decreased remarkably to $10.3 \text{ J kg}^{-1} \text{ K}^{-1}$ at 190 K. The shift of the peak

Fig. 6 Magnetic-entropy changes of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_x$ compounds at an external field change from 0 to 1.5 T for $x = 0$ and $x = 0.3$ **a** and from 0 to 5 T for $x = 1.2$ and $x = 2.1$ **b**

temperature to higher temperatures and the remarkable decrease in the maximal ΔS_m are ascribed to the higher T_C and the less pronounced first-order transition caused by nitrogenation. As stated above, $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ which consists of two 1:13 phases exhibits two transitions at 210 and at 295 K. The two transitions lead to a ΔS_m larger than $2 \text{ J kg}^{-1} \text{ K}^{-1}$ over the large temperature range from 200 to 310 K with a maximum ΔS_m value of $5.3 \text{ J kg}^{-1} \text{ K}^{-1}$ for a field change of 5 T at 225 K (as shown in Fig. 6b). For $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{2.1}$, the MCE has much decreased to a maximum ΔS_m of $2.0 \text{ J kg}^{-1} \text{ K}^{-1}$ for a field change of 5 T at 215 K which is due to the fact that a large amount of $\alpha\text{-Fe}$ has precipitated upon increasing the nitrogenation temperature to 650 °C.

4 Conclusions

$\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}$ nitrides with a particle size of 100–150 μm were prepared by using a self-made furnace in a high-purity N_2 atmosphere of 40 MPa. Curie temperature T_C of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{0.3}$ increases from 187 K for the parent bulk compound to 195 K. Moreover, the maximum hysteresis loss for a field change from 0 to 1.5 T at T_C is considerably reduced from 53 to 3 J kg^{-1} and a large ΔS_m of more than $10 \text{ J kg}^{-1} \text{ K}^{-1}$ is maintained. The XRD pattern of $\text{La}_{0.5}\text{Pr}_{0.5}\text{Fe}_{11.4}\text{Si}_{1.6}\text{N}_{1.2}$ exhibits two 1:13 phases and, accordingly, the magnetization exhibits two transitions at 210 and at 295 K. The two transitions lead, for a field change from 0 to 5 T, to a ΔS_m value larger than $2 \text{ J kg}^{-1} \text{ K}^{-1}$ over a large temperature range from 200 to 310 K with a maximum ΔS_m value of $5.3 \text{ J kg}^{-1} \text{ K}^{-1}$ at 225 K. The present nitrogenation study may be useful for the practical application of the $\text{La}(\text{Fe},\text{Si})_{13}$ -based compounds.

Acknowledgments This work was financially supported by the Liaoning Provincial Natural Science Foundation (No. 2013020105) and the Shenyang Science and Technology Foundation (No. F13-316-1-39).

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