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Magnetic properties and magnetocaloric effect in $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds

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ABSTRACT

The structural, magnetic and magnetocaloric properties in the cubic $NaZn_{13}$-type $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds with $x=1.2$, 1.4, 1.6 and 1.8 are reported. All of the investigated samples are ferromagnetically ordered. The Fe saturation moment at 5 K is independent of Si concentration, having a value of $2.05 \pm 0.09\ \mu_B$, which suggests a high degree of localization. The Curie temperatures increase almost linearly with the increase in silicon concentration. The large ΔS_M values obtained for samples with low Si content are mainly due to the first-order character of the transition at T_C. The field-induced transition above T_C causes an asymmetric broadening of the ΔS_M peak towards higher temperatures for higher applied fields. With increasing Si content, the first-order character of the transition at T_C is diminished, leading to lower ΔS_M values. The potential use of these materials in magnetic refrigeration is discussed.

1. Introduction

The magnetocaloric effect (MCE) displays itself as the production or absorption of heat by a magnetic material under the action of a magnetic field [1]. Recently, an increased interest has been observed in the MCE and magnetocaloric materials due to their practical application in refrigeration devices, especially due to the fact that a series of acting magnetic refrigerator prototypes have been developed, which have significant potential to be incorporated into the marketplace [2].

Among magnetic materials with high magnetocaloric properties, the $NaZn_{13}$-type $LaFe_{13-x}Si_x$ system has been suggested to be a good candidate for near-room-temperature magnetic refrigeration due to the high MCE values reported for these materials [1–14] and due to the fact that their transition temperature can be easily modified by adjusting the dopant concentration [3,6–8,11,12]. It was reported that $La(FeSi)_{13}$ compounds show isotropic Heisenberg ferromagnetic and anomalous critical behavior [15]. The $LaFe_{13}$ compound does not exist due to a positive heat of formation between La and Fe [5,7], however, a small amount of Si can stabilize the structure, leading to the formation of the pseudobinary compound $LaFe_{13-x}Si_x$ [2].

The compounds with low concentrations of Si exhibit a first-order magnetic phase transition from a ferromagnetic to a paramagnetic state and a large, discontinuous, negative lattice volume variation with no structural change at the Curie temperature [13,16]. A field-induced itinerant-electron metamagnetic (IEM) phase transition from a paramagnetic to a ferromagnetic state takes place for temperatures slightly above T_C [9,13], which causes an asymmetrical broadening of the $\Delta S_M(T)$ peak towards higher temperatures with increasing applied fields [13]. The large entropy change found in these materials was attributed to the first-order character of the transition at T_C [13]. Hu et al. [9] noted that the IEM transition above T_C causes a magnetic hysteresis above a critical field H_C. It was also reported that the critical field increases and that the magnetic hysteresis width narrows with increasing temperature [9]. With increasing Si content, the Curie temperature increases, however, the first-order nature of the transition at T_c diminishes [3,9,15] and changes to second-order for high concentrations of Si, leading to a smaller entropy change at the transition [3,14].

In recent papers, it was reported that low field hysteresis and easily tunable Curie temperatures can be achieved for $LaFe_{13-x}Si_x$ compounds through the addition of substitutional [3,7,11] or interstitial elements [3,8,12], or through new preparation methods [3,11]. The $La_{0.7}Nd_{0.3}Fe_{11.5}Si_{1.5}$ compound was successfully prepared and large magnetocaloric effect in moderate magnetic field was shown [17]. The magnetic behavior is similar with that of the compounds with Pr substitution on La site [18]. It was shown that a partial substitution with Co on Fe site lead to an increase of the transition temperature.

Considering the previous promising results obtained on $La_{0.7}Nd_{0.3}Fe_{11.5}Si_{1.5}$ compound it was our purpose to study the magnetic properties and the magnetic entropy change in $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds in a large composition range. Our results show clearly the changes in transition from a

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first-order magnetic transition to a second-order or a weak first-order magnetic transition. Large values were found for the magnetic entropy changes in fields of 0–2 T, field which can be produced by permanent magnets.

Based on the obtained results, the potential use of these materials in magnetic refrigeration is discussed.

2. Experimental

The $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ samples with $x=1.2$, 1.4, 1.6 and 1.8 were prepared by arc-melting of the constituent elements in a high-purity Ar atmosphere. The obtained buttons were remelted several times in order to ensure homogeneity. The samples were then wrapped in tantalum foil, sealed in a quartz glass tube under vacuum and annealed at 1400 K for 4 days.

X-ray measurements were carried out on powder samples at room temperature on a Bruker D8 Advance diffractometer using Cu $K\alpha$ radiation. The lattice parameters and phase percentages were calculated using the FullProf software. X-ray results confirm the formation of the $NaZn_{13}$ phase in all four samples after annealing and the presence of a small α-Fe impurity phase. Magnetic measurements were performed in the 4.2–300 K temperature range in external applied fields up to 12 T, using a vibrating sample magnetometer (VSM) made by Cryogenic Limited. The Curie temperature for each sample was determined from $M(T)$ measurements as the minimum in dM/dT. The isothermal magnetization curves were measured in magnetic fields from 0 to 4 T around the Curie temperature in 5 K steps. The saturation magnetizations, M_s, were determined from magnetization isotherms according to the law of approach to magnetic saturation:

$$M = M_s(1-b/H)+\chi_o H \quad (1)$$

where b is the coefficient of magnetic hardness and χ_o the Pauli-type contribution.

The magnetic entropy changes were determined from magnetization isotherms, between zero field and a maximum field (H_0) using the thermodynamic relation:

$$\Delta S_M(T,H_0) = S_M(T,H_0)-S_M(T,0) = \frac{1}{\Delta T}\int_0^{H_0}\left[M(T+\Delta T,H)-M(T,H)\right]dH \quad (2)$$

where ΔT is the temperature increment between measured magnetization isotherms ($\Delta T=5$ K for our data). The magnetic cooling efficiency was evaluated by considering the magnitude of the magnetic entropy change, ΔS_M and its full-width at half-maximum (δT_{FWHM}). The product of the ΔS_M maximum and $\delta T_{FWHM}=T_2-T_1$:

$$RCP(S) = -\Delta S_M \cdot \delta T_{FWHM} \quad (3)$$

is the so-called relative cooling power (RCP) based on the magnetic entropy change.

3. Results and discussions

The XRD patterns of $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds are presented in Fig. 1. In the hypothetical $LaFe_{13}$ structure, the La atoms occupy the 8*a* sites while the Fe atoms are located in the 8*b* and 96*i* sites. The location of Si atoms in the $LaFe_{13-x}Si_x$ lattice is not clear yet. Wang et al. [5] have shown, from neutron diffraction data, that Si is randomly distributed between the 8*b* and 96*i* sites. Later on it was suggested that Si atoms occupy mainly the 96*i* site [19]. From the analysis of the ^{57}Fe Mössbauer spectra Hamdeh and co-workers have concluded that Si atoms occupy the 96*i* sites only [20], in agreement with earlier neutron diffraction data obtained on an isomorphous compound with Al instead of Si

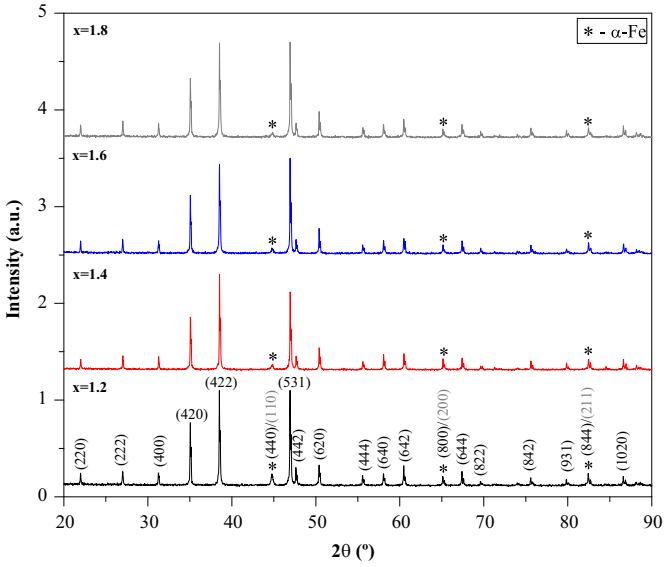

Fig. 1. X-ray diffraction patterns for the annealed $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds with $1.2 \le x \le 1.8$.

Table 1
Lattice parameters and phase concentrations in $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds.

x	a (Å)	$NaZn_{13}$ phase (%)	α-Fe phase (%)
1.2	11.45(9)	94	6
1.4	11.46(1)	95.9	4.1
1.6	11.45(7)	97.9	2.1
1.8	11.45(8)	98.8	1.2

[21], in which the same strong preference of Al atoms for 96*i* sites was evidenced. In our Rietveld analysis it was assumed that Si atoms occupy 96*i* site. All the $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ prepared compounds crystallized in a cubic $NaZn_{13}$-type structure. A small amount of *αFe* phase was also detected. The lattice parameters obtained from XRD patterns are weakly dependent on silicon content (see Table 1). It was found that the amount of *αFe* phase decrease when Si content increases being around 1.2% for the compound with $x=1.8$.

The temperature dependences of the magnetizations for the samples with $x=1.2$ and $x=1.6$ are presented in Fig. 2. The magnetizations were measured in heating and cooling processes under a magnetic field of 0.2 T. In all samples the presence of thermal hysteresis was evidenced, indicating the presence of a thermally induced first-order magnetic transition at Curie temperature (T_C). The thermal hysteresis was estimated from the difference in transition temperatures measured in the heating and the cooling processes. The thermal hysteresis decreases when silicon content increases from 9 K for the sample with $x=1.2$ to 5 K for the sample with $x=1.8$. This behavior implies that the increase of Si concentration in $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds can weaken the thermally induced first order magnetic transition at T_C as was previously reported for $La_{0.7}Pr_{0.3}Fe_{13-x}Si_x$ compounds [18]. The Curie temperatures were defined as the temperatures at which the dM/dT versus temperature curves measured during heating present a minimum. The T_C values increase linearly with silicon concentration from 181 K ($x=1.2$) to 213 K ($x=1.8$) (Table 2). Three types of interactions have to be considered: Fe–Fe, Nd–Fe and Nd–Nd. Usually, the rare-earth (R)–rare-earth interaction in iron rich R–Fe intermetallic compounds are

negligible [22] while the *R*–Fe interactions are weak comparing with Fe–Fe ones. In these compounds, the Curie temperature is determined mainly by the Fe–Fe and the R–Fe interactions but generally, the Fe–Fe interactions are dominant. There are two types of Fe–Fe exchange interactions according to Givord and Lemaire [23], a positive and a negative one, depending on the Fe–Fe interatomic distance. The interaction is positive if the distance between iron atoms is larger than 0.245 nm and negative if the distance is smaller. The distance between iron pairs in our compounds is larger between the atoms situated on 96*i* site (approx. 0.256 nm) than between iron atoms located on different sites (approx. 0.245 nm) [5]. Two effects have to be considered by replacing Fe with Si. The first one is the hybridization between the *sp* orbitals of Si and the *d* orbit of Fe [19]. It was shown that the density of states (DOS) of the Fe-3*d* down spin states consists of two prominent peaks and the Fermi energy is pinned at the deep valley between them, which reflects that this electronic system is stabilized with the help of spin polarization [19]. The second one is the lattice contraction which leads to a reduction in T_C [24,25]. Han and Miller [26] have shown that the contacts between iron atoms shorter than 0.245 nm decreases upon increasing silicon content, accompanied by a reduction of the effect of antiferromagnetic Fe–Fe exchange interaction and leads to an increase in the Curie temperature. The enhancement of the Curie temperature can be described in the spin fluctuation theory in terms of changes in the electronic structure [27]. It was shown that the increase of Curie temperatures due to substitutional modifications can be explained by a decrease in spin fluctuations, which is caused by lowering the DOS at the Fermi level upon substitution [27]. In our compounds the lattice parameters are little dependent on silicon content. The increase of T_C with increasing Si concentration in $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds can be attributed to a decrease of the total DOS at the Fermi level caused by the substitution of Si for Fe as was previously reported for $LaFe_{13-x}Si_x$ compounds.

The magnetization isotherms, in external magnetic fields up to 12 T, are presented in Fig. 3. All the samples have similar behavior, saturation being already attended in field of 2 T. The compounds are ferromagnetically ordered, the neodymium and iron magnetic moments being parallelly oriented. The saturation moments at 5 K were determined to be 26.3 μ_B/f.u. ($x=1.2$) and decrease with increasing silicon content to 23.7 μ_B/f.u. for $x=1.8$. From these values, first, was extracting the *αFe* contribution (we assume a moment of 2.2 μ_B/Fe atom like in bulk iron). We have to note that it was shown previously, by band structure calculations, that La has magnetic moment in $LaFe_{13-x}Si_x$, antiparallel oriented with Fe and Nd moments and decrease from 0.5 μ_B/atom (hypothetically $x=0$ compound) to 0.39 μ_B/atom ($x=2$) [26].

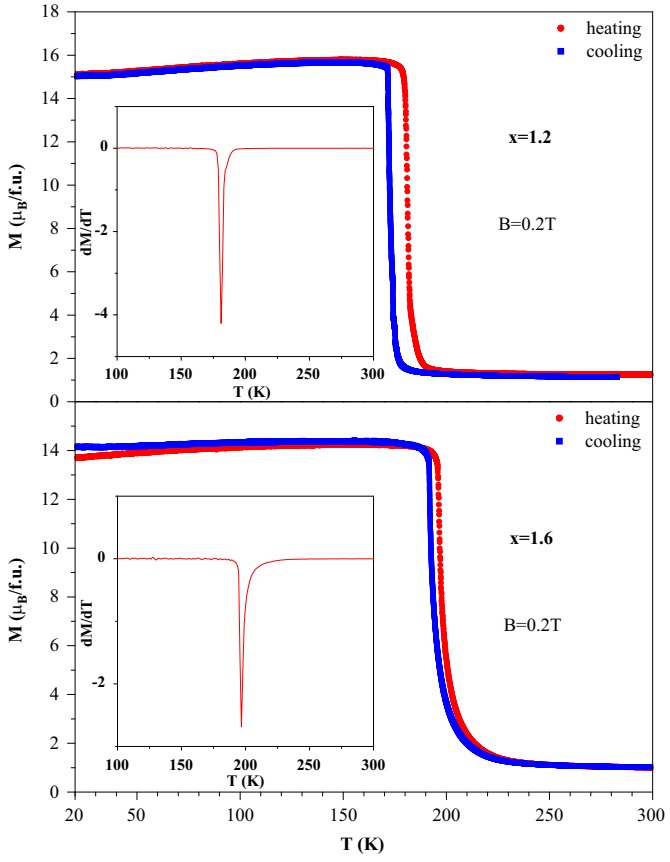

Fig. 2. The temperature dependences of the magnetizations for $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds with $x=1.2$, respectively, $x=1.6$ measured in a low magnetic field of 0.2 T, during heating and cooling. The insets represent the derivative of magnetization versus temperature.

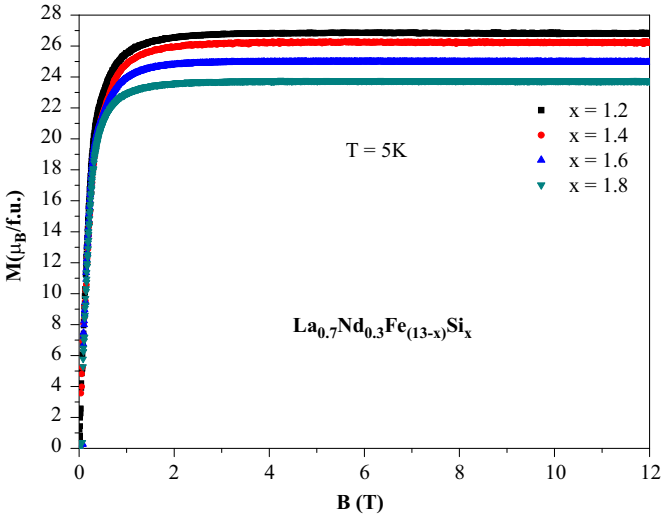

Fig. 3. The magnetization isotherms measured in external magnetic fields up to 12 T.

Table 2
Curie temperatures, maximum entropy changes in external magnetic fields 0–2 T, respectively, 0–4 T, RCP(ΔS) and RCP(ΔS)/ΔB values for $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds.

x	T_C (K)	$-\Delta S_{M(max)}$ $B=0$–4 T (J/kg K)	RCP(ΔS) $B=0$–4 T (J/kg)	RCP(ΔS)/ΔB $B=0$–4 T (J/kg T)	$-\Delta S_{M(max)}$ $B=0$–2 T (J/kg K)	RCP(ΔS) $B=0$–2 T (J/kg)	RCP(ΔS)/ΔB $B=0$–2 T (J/kg T)
1.2	181	28.7	435	109	27	191	96
1.4	188	26.6	423	106	19.4	200	100
1.6	197	17.2	331	84	13.2	153	76
1.8	213	12.6	277	69	7.9	136	68

Probably, the magnetic moment of La has values in the range 0.45–0.38 μ_B/atom in our compound. A lanthanum contribution of approx. -0.28 μ_B/atom can be assumed to the total magnetic moment, which will modify the average Fe magnetic moment with 0.02 μ_B/atom. Considering that the Nd magnetic moment is the same as in the Nd^{3+} ion the iron magnetic moment was determined. It was found to be quite independent on composition, being 2.05 ± 0.09 μ_B/Fe atom, which show a high degree of localization.

The magnetization isotherms for the samples with $x=1.4$ and 1.8 measured in a wide temperature range around the transition temperatures with a step of 5 K are shown in Fig. 4. One can see that for the sample with $x=1.4$ an itinerant electronic metamagnetic transition is present in the paramagnetic region above the Curie temperature. We note that a negative lattice volume variation with no structural change was reported for similar compounds with low silicon content [13,26]. The simultaneously sharp change of magnetization and lattice parameters at the transition temperature should strongly influence the magnetic entropy change. The increase of silicon content weakens the IEM transition and in the case of the sample with $x=1.8$ the IEM transition is not present. This behavior is confirmed by the presence of an obvious inflexion point in the Arrott plots at T_C, which is the signature of the IEM transition from paramagnetic to ferromagnetic order above T_C (Fig. 5). No inflexion point at the transition temperature was found in the $La_{0.7}Nd_{0.3}Fe_{11.2}Si_{1.8}$ sample. Characteristics of a second-order type transition or a weak first-order magnetic transition were found in the sample with $x=1.8$ while the compounds with lower silicon content undergo a first-order magnetic phase transition at T_C. The presence of the thermal hysteresis at transition in sample with $x=1.8$ show that the second-order phase transition is not pure.

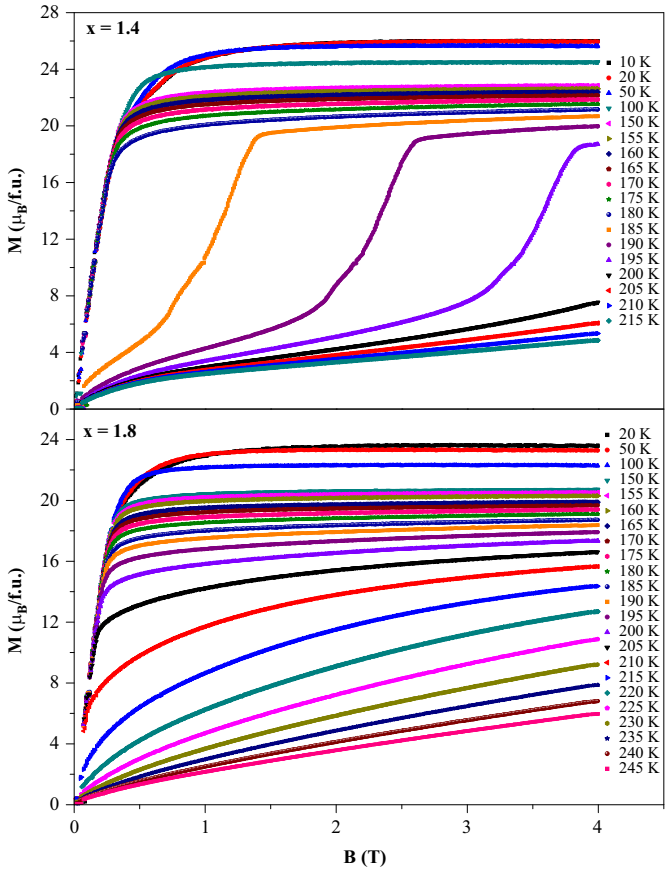

Fig. 4. The magnetization isotherms for $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds ($x=1.4$ and 1.8) measured around the transition temperatures with a step of 5 K.

No magnetic hysteresis above the transition temperature was found for external magnetic fields lower than 2 T in all cases, as it is expected for the paramagnetic region. In the case of the compounds which present an IEM transition the magnetic hysteresis can be seen at higher magnetic fields. Magnetic hysteresis loops for the compounds with $x=1.2$ and 1.8 are shown in Fig. 6.

The magnetic entropy change was calculated from magnetization isotherms using the Maxwell relation. The magnetic entropy changes as function of temperature for different magnetic field changes are presented in Fig. 7. The obtained maximum entropy change values were found to decrease from 28.7 J/kg K for $x=1.2$–12.6 J/kg K for $x=1.8$ for a magnetic field change from 0 to 4 T. The large ΔS_M values obtained for samples with low Si content are mainly due to the first-order character of the transition at T_C. Also, the field-induced transition above T_C causes an asymmetric broadening of the ΔS_M peak towards higher temperatures for higher applied fields. With increasing Si content, the first-order character of the transition at T_C is diminished, leading to lower ΔS_M values. It is worthwhile to note that the maximum entropy change decrease only to 27 J/kg K ($x=1.2$) and 7.9 J/kg K ($x=1.8$) respectively, in external magnetic fields of 0–2 T, which is very important for magnetic refrigeration.

A characteristic parameter for magnetocaloric materials, is the relative cooling power. A large $RCP(\Delta S)$ corresponds to a better magnetocaloric material. The maximum values of the magnetic entropy changes determined in external magnetic fields between 0 and 2 T, respectively, 0–4 T are presented in Table 2. It was also suggested that for characterizing magnetocaloric materials, it is more convenient to use the specific renormalized cooling power, defined as $RCP(\Delta S)/\Delta B$, relative to the external field variation ΔB [1]. In the present system, high $RCP(\Delta S)$ values were shown, due to the large maximum values of the magnetic entropy changes. As an example, for compounds with $x=1.2$ and 1.8 $RCP(\Delta S)/\Delta B$ values of the order of 109 J/kgT and 69 J/kgT, respectively, were obtained. These values are comparable with the results obtained on the best magnetocaloric materials such as pure Gd (111 J/kgT) [28], $La_{0.7}Pr_{0.3}Fe_{11.5}Si_{1.5}$ (105 J/kgT) [24], $MnFeP_{0.65}As_{0.35}$ (82 J/kgT) [29] and $Gd_5Ge_{1.8}Si_{1.8}Sn_{0.4}$ (31 J/kgT) [30].

We note that determined $|\Delta S|$ values are only approximate. These are influenced by the rate of increasing magnetic field, during the measurements as well as by the approximation used in determining $|\Delta S|$ values from discrete sets of magnetization data [31,32]. The entropy changes are also sensitive to the magnetic hardness of the material.

4. Conclusions

The $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds were prepared. A small amount of αFe phase was also detected. All compounds are ferromagnetically ordered. The iron magnetic moment at 5 K is quite independent on composition, being 2.05 ± 0.09 μ_B/Fe atom, which suggests a high degree of localization. The Curie temperatures increase almost linearly with the increase of silicon concentration. This behavior was attributed to a strong s–d orbital hybridization caused by the substitution of Si for Fe. An itinerant electronic metamagnetic transition is present in the paramagnetic region above the Curie temperatures in the samples with low silicon content. The increase of silicon content weakens the IEM transition and in the case of the sample with $x=1.8$ the IEM transition is not present. This behavior was confirmed by the presence of an obvious inflexion point in the Arrott plots at T_C. Characteristics of a second-order type transition or a weak

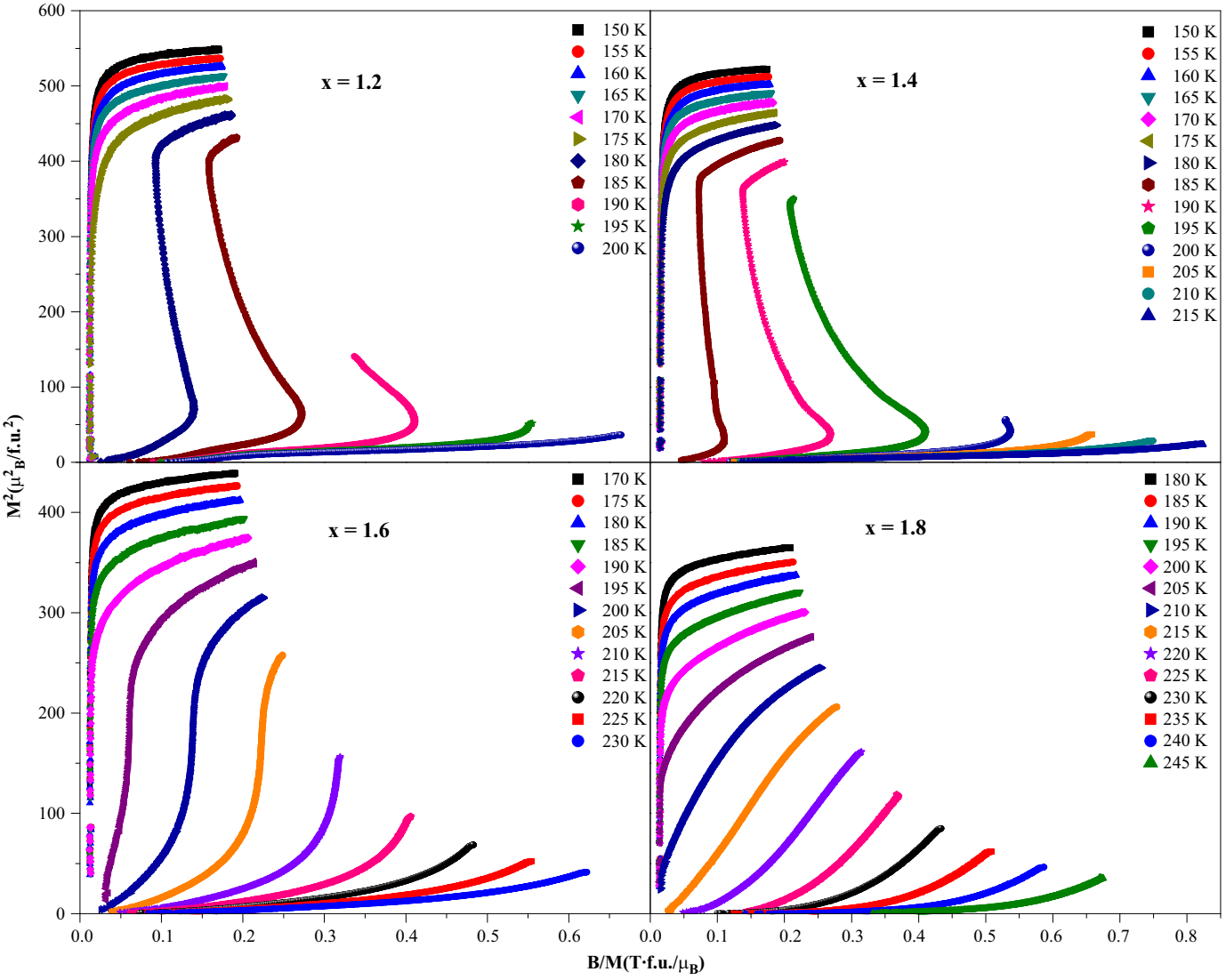

Fig. 5. Arrott plots for the $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds.

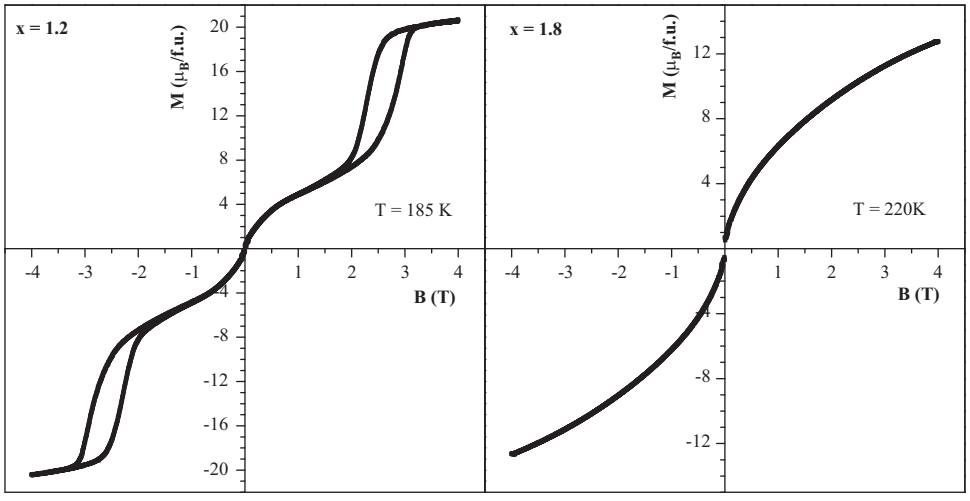

Fig. 6. Magnetic hysteresis loops for the $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$ compounds with $x=1.2$ and 1.8.

first-order magnetic transition were found in the sample with $x=1.8$ but the presence of the thermal hysteresis at transition show that the second-order phase transition is not pure.

The increase of Si concentration causes a reduction of the magnetic entropy change due to the weakening of the IEM transition. The relative cooling power values are high enough in

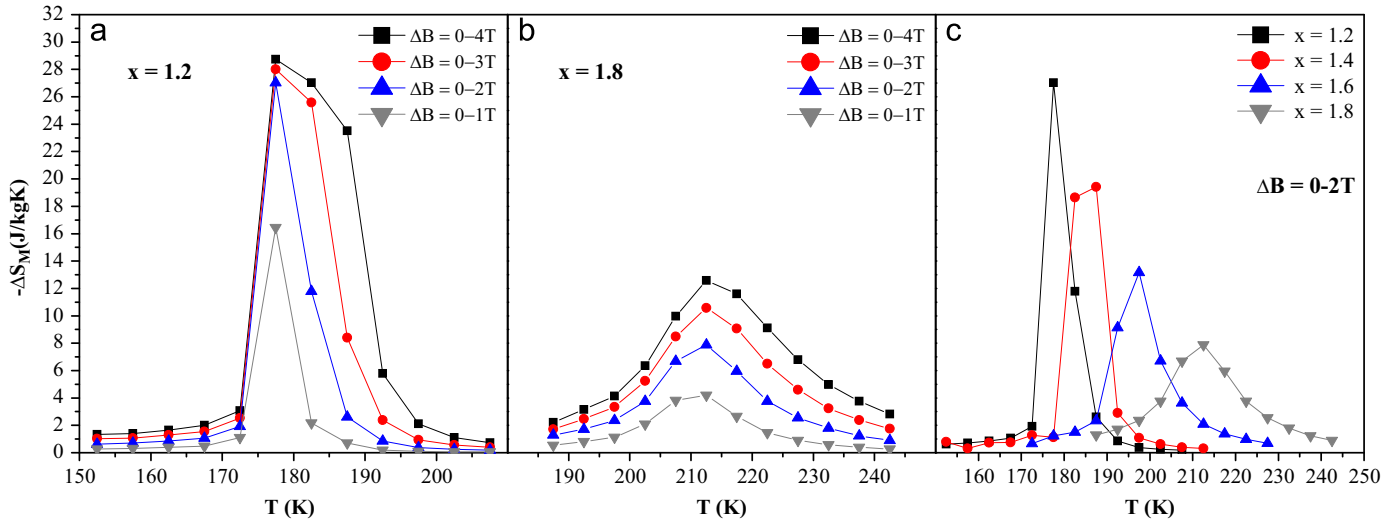

Fig. 7. The magnetic entropy changes as function of temperature in different external magnetic fields for the samples: (a) $La_{0.7}Nd_{0.3}Fe_{11.8}Si_{1.2}$; (b) $La_{0.7}Nd_{0.3}Fe_{11.2}Si_{1.8}$; and (c) $La_{0.7}Nd_{0.3}Fe_{13-x}Si_x$.

order to consider this compounds as possible candidates as refrigerant materials.

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