

Role of lattice contraction in the magnetocaloric effect in $\text{LaFe}_{11.5}\text{Si}_{1.5}$

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Abstract

The magnetocaloric effect (MCE) and metamagnetic transition of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ compound are studied by the measurement of magnetization, ac susceptibility, and heat capacity. The ferromagnetic order temperature is observed to be 196 K. The field-induced metamagnetic transition that causes a paramagnetic-to-ferromagnetic transition takes place above 196 K. Although it is fairly strong, the MCE in $\text{LaFe}_{11.5}\text{Si}_{1.5}$ is much weaker than expected in theory. Possible mechanisms are discussed based on the thermodynamics theory. It is possible that the magnetic entropy change is counteracted by the lattice entropy change resulting from the lattice contraction of the compound at the magnetic transition.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Materials with large magnetocaloric effect (MCE) are widely investigated for their potential applications as favourable refrigerants. MCE means isothermal entropy change or adiabatic temperature change through magnetizing or demagnetizing magnetic solids. This process is similar to compressing or expanding gas. But compared with the process of compressing gas, the magnetic refrigeration technique based on MCE is friendly to the environment. It also has high energy efficiency. Investigations of MCE are nowadays focused on materials which have gone through a first-order phase transition [1–6]. It is believed that the isothermal entropy change or adiabatic temperature change should be prominent in the vicinity of phase transition.

The lattice cell, however, always undergoes a discontinuous change accompanying a first-order magnetic phase transition. This lattice effect actually could not be ignored in view of the physics and the application. For example, the lattice cell of the $\text{LaFe}_{13-x}\text{Si}_x$ inter-metallic compounds shows a discontinuous negative expansion (ferromagnetic state to paramagnetic state) which is large, and approaches 4% accompanying a first-order phase transition [1]. Therefore, the $\text{LaFe}_{13-x}\text{Si}_x$ inter-metallic compounds are good candidates for investigating the influence of the lattice effect on the MCE.

$\text{LaFe}_{13-x}\text{Si}_x$ inter-metallic compounds have the NaZn_{13} -type cubic structure with the space group $Fm\bar{3}c$, and stabilize in the composition range between $x = 1.56$ and 2.47 [7]. Lanthanum atoms occupy 8a sites, while iron atoms and silicon atoms occupy 8b sites and 96i sites randomly [8]. $\text{LaFe}_{13-x}\text{Si}_x$ compounds in the ground state are ferromagnetic and have high magnetization. With increasing silicon content, the moments of Fe atoms decrease and the Curie temperature increases, which is usually found in Invar-type alloys, as pointed out by Palstra *et al* [7], and the phase transition changes from the first order to the second order. It is pointed out by Fujita *et al* that $\text{LaFe}_{13-x}\text{Si}_x$ compounds show a field-induced first-order phase transition from the paramagnetic state to the ferromagnetic state, which is called the itinerant electron metamagnetic (IEM) transition [9–13]. A large MCE [1–3] and a magnetovolume effect [1, 2, 9, 10, 12, 13] resulting from an IEM transition have been observed in $\text{LaFe}_{13-x}\text{Si}_x$ compounds. As for $\text{LaFe}_{11.4}\text{Si}_{1.6}$ compound, the value of the entropy change, $|\Delta S|$, reaches $19.4 \text{ J kg}^{-1} \text{ K}^{-1}$ at 0–5 T [1]. A large value approaching $30 \text{ J kg}^{-1} \text{ K}^{-1}$ in $\Delta H = 5 \text{ T}$ has been observed in $\text{La}(\text{Fe}_{0.88}\text{Si}_{0.12})_{13}$ and its hydrides. The Curie temperature of the hydrides is set between 195 and 336 K, with the entropy change being approximately unaffected [3]. A strong magneto-volume effect [1, 2, 9, 10, 12, 13] exists in $\text{LaFe}_{13-x}\text{Si}_x$ compounds, which makes the Curie temperature very sensitive to the lattice parameter. Atoms of hydrogen enter into the interstitial sites and enlarge the lattice parameter. As a result, the Curie temperature increases remarkably, though the moments of magnetic atoms have almost no change. There is an opinion that the density of state (DOS) curve near the Fermi level is hardly affected by the method of hydrogen absorption and the characteristics of IEM remain [3] which is why the Curie temperature is set from 195 to 336 K and the large MCE is preserved. However, the mechanisms for the lattice cell change accompanying the metamagnetic transition and its effect on the total entropy change are still unclear.

The MCE due to the metamagnetic transition is very often estimated using the Maxwell relation or the heat capacity method. According to the thermodynamics, either the Maxwell relation or the heat capacity method only gives the total entropy change if magneto-elastic coupling exists in a magnetic solid. As a complicated system, the total entropy is composed of an electronic part, a magnetic part, and a lattice part etc. $\text{LaFe}_{13-x}\text{Si}_x$ compounds [1, 2, 10, 12] and its hydrides [13] show discontinuous lattice contraction at the first-order phase transition. This implies that magneto-elastic coupling should exist and that the lattice entropy change accompanying the phase transition should be obvious. An effective and accurate method for estimating the different parts of total entropy in a real material is necessary for a study of the MCE and the application of the magnetic refrigeration technique. Unfortunately, this kind of work is deficient.

In this paper the temperature dependence of the dc and ac susceptibilities under various fields, the isothermal magnetization and the heat capacity under different fields have been measured. The total entropy change and magnetic entropy change have been investigated by using the Maxwell relation, the heat capacity method and the mean-field approximation (MFA). Possible explanations for the discrepancy between the experimental and theoretical results have been given.

2. Experiment

A $\text{LaFe}_{13-x}\text{Si}_x$ sample with the normal composition $x = 1.5$ was prepared by arc-melting the appropriate amounts of raw materials in an ultra-pure argon gas atmosphere. A 5% excess of lanthanum over the stoichiometric composition was taken. The purity is 99.9% for lanthanum and iron, and 99.99% for silicon. The product was sealed in a high-vacuum quartz tube, and a post-annealing at 1323 K for 15 days was performed for homogenization. The single-

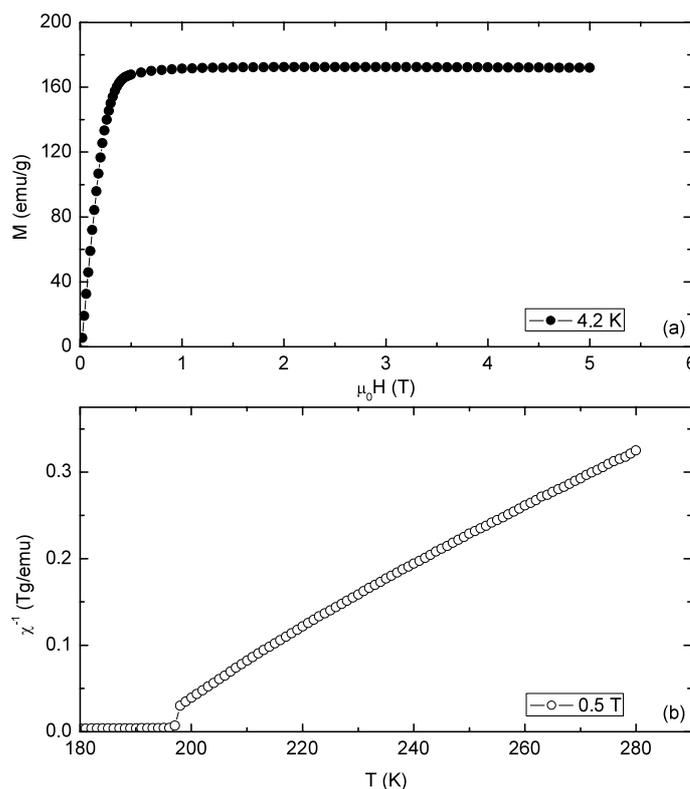


Figure 1. The magnetization at 4.2 K (a) is represented by the solid circle symbols; the temperature dependence of dc reverse magnetic susceptibility at 0.5 T (b) is represented by the hollow circle symbols. The solid lines are guides for the eye.

phase specimen with NaZn_{13} -type structure was confirmed by x-ray powder diffraction study. The magnetization and heat capacity measurements were conducted on a commercial physical properties measurement system (PPMS, Quantum Design).

3. Results and discussions

3.1. Magnetocaloric effect due to metamagnetism transition

Devonshire has demonstrated a first-order phase transition induced by the external field or heating based on an analysis of the thermodynamics potential expanded in the order parameter [14]. The interest evoked in the IEM transition can be cast back to the works on the Co-based Laves phase and pyrite compounds, which have been investigated theoretically and experimentally [15, 16]. The metamagnetism transition, however, appears not only in an itinerant electronic magnet but also in a local moment magnet such as MnAs [5]. As for $\text{LaFe}_{13-x}\text{Si}_x$ compounds, the high magnetization and metamagnetic transition are regarded as marks of possessing a large MCE. The temperature dependences of dc and ac susceptibilities are presented in figures 1 and 2. The $\chi^{-1}-T$ relation in the paramagnetic state obeys the Curie–Weiss law (see figure 1(b)). The upward shift of the Curie temperature with increasing dc magnetic field is one of the features of the metamagnetic transition, which can be seen in figure 2. The slope of the Curie temperature versus the external magnetic field is about 4 K T^{-1} .

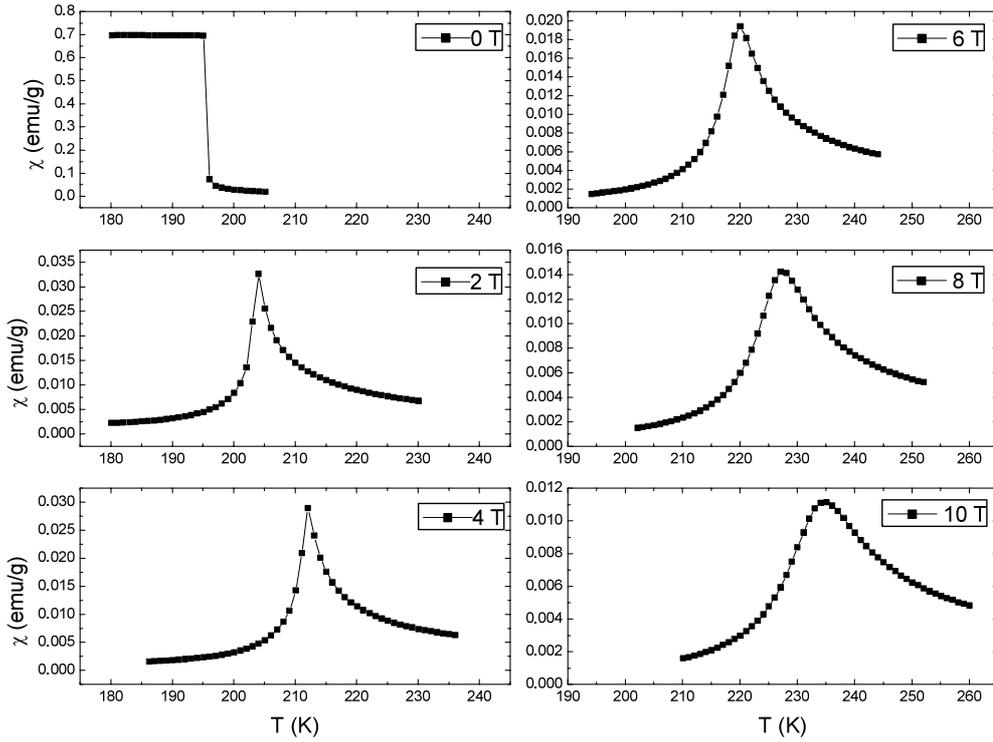


Figure 2. The ac magnetic susceptibility measured under different dc magnetic fields with an ac magnetic field of 10 Oe and a frequency of 1 kHz. The solid lines are guides for the eye.

With increasing field, the Curie temperature is driven to higher temperature. Figure 3 shows the isothermal magnetization curves. The critical field presents a linear change with temperature increasing and the slope is $\sim 0.25 \text{ T K}^{-1}$ (see inset to figure 3). The magnetizations change discontinuously in the range of a few kelvin above the phase transition temperature 196 K, but change continuously at higher temperatures. The phase transition should disappear when the magnetic field is strong enough, according to the Devonshire theory [14]. This kind of character can be seen in figures 2 and 3 in the very strong field condition where the curves change continuously with increasing temperature or magnetic field.

The metamagnetic transition of $\text{LaFe}_{13-x}\text{Si}_x$ causes a large MCE (large total entropy change), which is estimated by using the Maxwell relation. However, the extent of the magnetic entropy change accompanying the metamagnetic transition is still unclear. In the Rhodes–Wohlfarth plot [17], the ratio of q_c/q_s can scale the itinerant electron feature, where $q_c = gJ$ is deduced from the Curie–Weiss constant, and $q_s = gJ$ is obtained from the low-temperature saturation magnetization [17–19]. The low-temperature saturation magnetization in the ferromagnetic state and the dc susceptibility in the paramagnetic state are shown in figures 1(a) and (b), respectively. The total angular momentum deduced from the dc susceptibility and low-temperature saturation magnetization are 1.5 and 1, respectively. The ratio $q_c/q_s \cong 1.31$ is near the value of Co ($q_c/q_s = 1.34$, $T_C = 1393 \text{ K}$). According to Moriya’s theory [19], when the Curie–Weiss law is held well and the Rhodes–Wohlfarth ratio is much larger than 1, the mean-square amplitude of the local spin fluctuation, S_L^2 , should increase dramatically with temperature. Considering $T_C = 196 \text{ K}$, the ratio q_c/q_s of $\text{LaFe}_{13-x}\text{Si}_x$

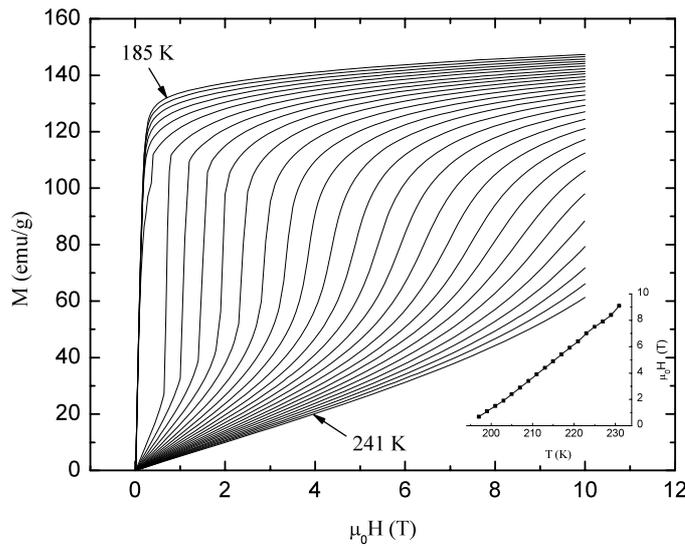


Figure 3. Isothermal magnetization from 0 to 10 T; the inset shows the critical field change with the temperature. The temperature changes from 185 to 241 K in steps of 2 K.

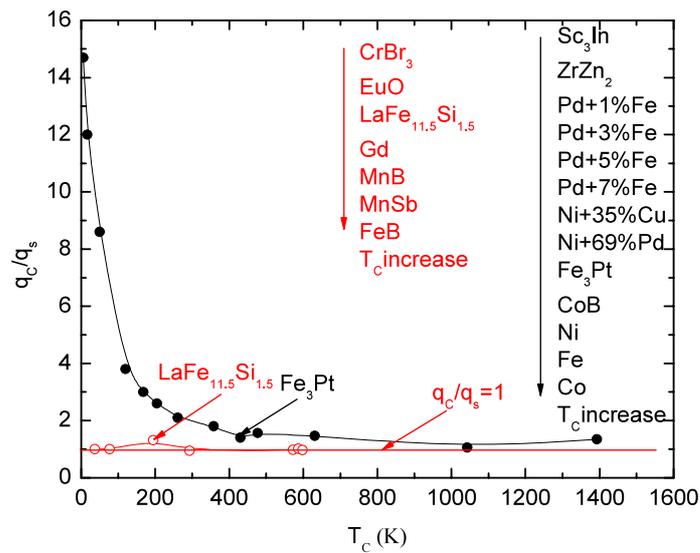


Figure 4. Rhodes–Wolfarth plot. Data, except for $\text{LaFe}_{11.5}\text{Si}_{1.5}$, are derived from [17] and [19].

shown in figure 4 is much nearer to the pure local electron ferromagnetism line compared with the itinerant electron line in the Rhodes–Wohlfarth plot. It is implied that the mean-square amplitude of the local spin fluctuation increases indistinctly with temperature. Therefore, the mean field is a reasonable approximation. Tishin [20] successfully studied the MCE of Fe, Co and Ni with the MFA. So the MFA is also adopted for effectiveness and convenience to estimate the magnetic entropy change in this paper. In the MFA, the magnetic entropy of the system can

be written as:

$$S_m = Nk_B \left[\ln \frac{\sinh \frac{2J+1}{2J}x}{\sinh \frac{x}{2J}} - x B_J(x) \right], \quad (1)$$

where N is the number of magnetic spins, $B_J(x)$ is the Brillouin function with $x = gJ\mu_B(H + H_m)/k_B T$, $g = 2.22$ is the Landé g factor, μ_B is the Bohr magneton, $J = 1$ is the total angular momentum which is attained from the low-temperature saturation magnetization curve, k_B is the Boltzmann constant, and H and H_m are the external magnetic field and molecular field, respectively. According to the relation between the molecular field coefficient and the Curie temperature, we can write x as:

$$x = \mu_s \left[H + \frac{3Jk_B T_C \sigma}{(J+1)\mu_s} \right] / k_B T. \quad (2)$$

The normalized magnetization $\sigma = M/N\mu_s$ with $\mu_s = gJ\mu_B$. The magneto-elastic coupling is introduced to the calculation through the parameter T_C , since the variation in T_C with lattice parameter is the outward manifestation of magneto-elastic coupling. In fact, T_C in equation (2) is determined by the minimum of the temperature dependence of magnetization. The Gibbs free energy should be determined from the pressure, magnetic field and temperature because of the strong magneto-elastic coupling. So it is easy to obtain the Maxwell relations:

$$\left(\frac{\partial V}{\partial H} \right)_{P,T} = - \left(\frac{\partial M}{\partial P} \right)_{H,T}, \quad (3)$$

$$\left(\frac{\partial V}{\partial T} \right)_{P,H} = - \left(\frac{\partial S}{\partial P} \right)_{T,H}, \quad (4)$$

$$\left(\frac{\partial M}{\partial T} \right)_{H,P} = \left(\frac{\partial S}{\partial H} \right)_{T,P}, \quad (5)$$

where V is volume and P is pressure. The total entropy change, in different magnetic fields at constant pressure, can be estimated by using equation (5) and written as:

$$S(H_2, T) - S(H_1, T) = \int_{H_1}^{H_2} \left(\frac{\partial M(H, T)}{\partial T} \right)_H dH. \quad (6)$$

The total entropy change also can be calculated from heat capacity data:

$$S(T, H) - S_0 = \int_0^T \left(\frac{C(T, H)}{T} \right)_H dT, \quad (7)$$

where S_0 is the entropy at $T = 0$ K, and is assumed to be zero. Figure 5 shows the magnetic entropy change obtained from equations (1) and (2), and the total entropy change determined from equation (6). It might be difficult to obtain the exact spontaneous magnetization by simply extrapolating high-field data to $H \rightarrow 0$ T because of the field-induced metamagnetic transition. So this paper focuses on the magnetic entropy change above the saturating field (2 T). The utmost magnetic entropy of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ is $Nk_B \ln(2J+1) = 125.60 \text{ J kg}^{-1} \text{ K}^{-1}$. It is interesting that the width of the half-maximum of the ΔS peaks calculated by using the MFA method are consistent with the results of the Maxwell relation, but the peaks of the MFA method are larger than the Maxwell relation method: $20.0 \text{ J kg}^{-1} \text{ K}^{-1}$ at 2–3 T, $26.0 \text{ J kg}^{-1} \text{ K}^{-1}$ at 2–5 T, $29.7 \text{ J kg}^{-1} \text{ K}^{-1}$ at 2–7 T, and $32.4 \text{ J kg}^{-1} \text{ K}^{-1}$ at 2–9 T. The magnitudes of the peaks attained by the Maxwell relation method are less than half of the MFA estimation. Obvious discrepancies exist over the whole ferromagnetic region, which may be a clue to the strong magneto-elastic coupling. The heat capacity in different magnetic fields shown in figure 6

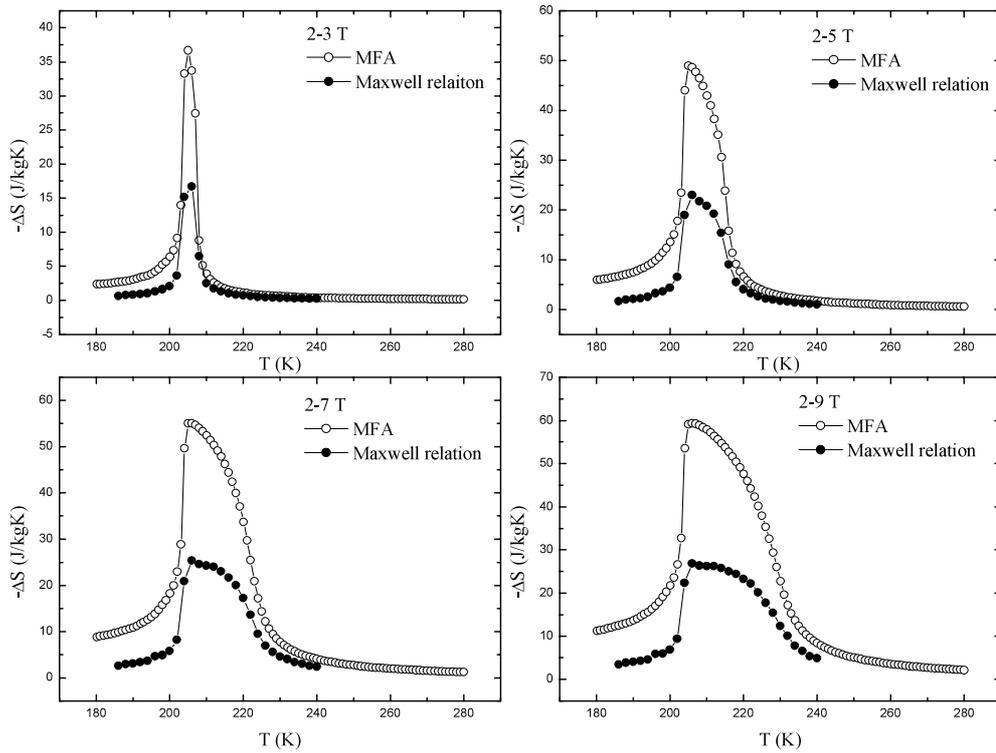


Figure 5. Temperature dependence of magnetic entropy change estimated by using the MFA (empty dots) and total entropy change calculated by using the Maxwell relation (solid dots) in variable magnetic field changes. The solid lines are guides for the eye.

is used to confirm the discrepancy between the theoretical estimations and the measurement results. The total entropy change determined by using equation (7) is achieved when the magnetic field changes from 2 to 5 T. The results attained from three methods are shown in figure 7. The peaks attained by using the MFA, the Maxwell relation and the heat capacity are 49.0 , 23.0 and $15.6 \text{ J kg}^{-1} \text{ K}^{-1}$, respectively. The difference between the magnetic method and the calorimetric method is obvious, but it can be attributed to the different measurement conditions [21]. The discrepancy between the magnetic entropy (calculated by using the MFA) and the total entropy (estimated by using the Maxwell relation and the heat capacity) looks to be in conflict with the original expectation that the magnetic entropy dominate the total entropy in this kind of first-order magnetic phase transition. Subsequently, the reasons will be revealed.

3.2. A viable explanation for the discrepancy between theory and experiment

The large MCE of $\text{LaFe}_{13-x}\text{Si}_x$ [1–3] has been reported before, but some phenomena were ignored. The magnitude of MCE is much lower than expected. It is well known that the large volume deformation is always accompanied by a first-order magnetic phase transition. The discontinuous negative lattice cell expansion of $\text{LaFe}_{13-x}\text{Si}_x$ is about 4% in the vicinity of a magnetic phase transition [1]. This is another clue to the strong magneto-elastic coupling. The mechanism is still blurry for why these kinds of magneto-elastic couplings affect the entropy change of the whole system. As a complex magnetic solid, the total entropy is approximately composed of an electronic part, a magnetic part, and a lattice part etc. But it is difficult to

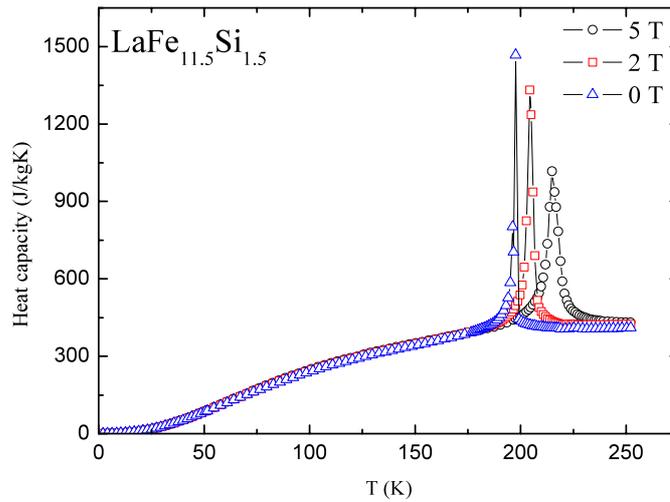


Figure 6. The heat capacity as a function of temperature and magnetic field from 2 to 252 K. The solid lines are guides for the eye.

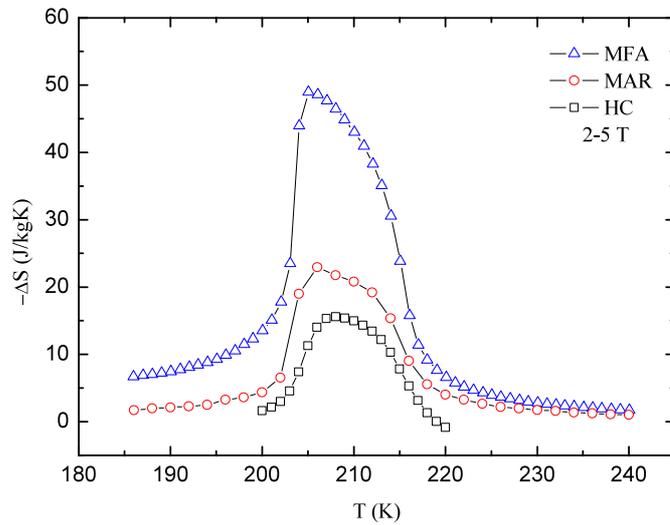


Figure 7. Temperature dependence of the magnetic entropy change estimated by using the MFA, total entropy change calculated by using the Maxwell relation (MAR) and heat capacity (HC). The solid lines are guides for the eye.

distinguish one from the other when all of them or part of them are coupling, especially in a first-order phase transition where the lattice changes obviously. Bean and Rodbell [22] have proposed a simple relation, $T_C = T_0(1 + \beta\omega)$, to explain the magneto-volume effect in MnAs, where T_C and T_0 are the Curie temperature and the Curie temperature in the absence of the lattice deformation respectively, β is the slope of the dependence of T_C on volume, and ω is the volume deformation. Recently, the Bean–Rodbell model is further developed to describe the influence of volume deformation on the giant MCE in $\text{Gd}_5(\text{Si}_x\text{Ge}_{1-x})_4$ [23], the colossal MCE in MnAs [24], and $\text{Mn}_{1-x}\text{Fe}_x\text{As}$ compounds [25]. Those results show that

the lattice entropy may enhance the entropy change and lead to colossal MCE. However, the studies on $\text{La}_{1-x}\text{Pr}_x\text{Fe}_{11.5}\text{Si}_{1.5}$ show a different viewpoint. The coexistence of paramagnetic and ferromagnetic phases may be responsible for the huge peak in the entropy change, always observed in colossal MCE, which is easily mistaken [26]. It is necessary to reveal the real function of the lattice in the MCE problem in the vicinity of the phase transition.

The stress can be looked on as the elasticity limit because of the reversible change of $\text{LaFe}_{13-x}\text{Si}_x$ volume deformations. Considering the spontaneous lattice cell negative expansion in the vicinity of the phase transition, we can assume that the stress should possess the *nonlinear* form:

$$F = -(1 + B\omega)K\omega. \quad (8)$$

The elastic energy per unit volume can be written as:

$$U = \frac{1}{2}K\omega^2 \left(1 + \frac{2}{3}B\omega\right). \quad (9)$$

The case of $\text{LaFe}_{13-x}\text{Si}_x$ can be discussed in the frame of thermodynamics. The Gibbs free energy per unit volume can be written as:

$$G = -HN\mu_s\sigma - \frac{3}{2} \left(\frac{J}{J+1} \right) Nk_B T_c \sigma^2 + \frac{1}{2}K\omega^2 + \frac{1}{3}BK\omega^3 + P\omega - TS, \quad (10)$$

where the first term on the right is the field term; the second is exchange; the third and the fourth are distortion; the fifth is pressure; and the last is entropy. The volume deformation $\omega = (V - V_0)/V_0$, where V and V_0 are the volume with and without the exchange interactions, respectively. Here V_0 is the volume of the paramagnetic state in zero external field and pressure. K is the modulus of compression, which is always positive, and B is a positive coefficient. Usually, the electronic entropy change is so small that it is negligible [27]. As for a first-order phase transition, the Gibbs free energy of the low-temperature phase (l) is equal to the high-temperature phase (h) at the critical point. According to the equation (10), we get:

$$T_c(S_h - S_l) = HN\mu_s(\sigma_l - \sigma_h) + \frac{3}{2} \left(\frac{J}{J+1} \right) Nk_B T_c(\sigma_l^2 - \sigma_h^2) + P(\omega_h - \omega_l) + \frac{1}{2}K(\omega_h^2 - \omega_l^2) + \frac{1}{3}BK(\omega_h^3 - \omega_l^3). \quad (11)$$

This always possesses $\sigma_h < \sigma_l$ and $|\omega_h| < |\omega_l|$, within the elasticity limit and in the condition of nonzero field and pressure:

- (a) When the lattice undergoes a negative expansion (ferromagnetism FM to paramagnetism PM) $\omega > 0$.

The sum of the last three terms in equation (11) is always negative, so the contributions of the lattice entropy change counteract the magnetic part.

- (b) When the lattice undergoes a positive expansion (FM to PM) $\omega < 0$.

If the sum of the last three terms in equation (11) is positive, the contributions of the lattice entropy change add to the magnetic part; if the sum of the last three terms in equation (11) is negative, the contributions of the lattice entropy change counteract the magnetic part.

In the conditions of zero field and high vacuum, $H \cong 0$, $\sigma_h \cong 0$, $P \cong 0$, and $\omega_h \cong 0$. Then equation (11) can be simplified as:

$$T_c(S_h - S_l) = \frac{3}{2} \left(\frac{J}{J+1} \right) Nk_B T_c \sigma_l^2 - \frac{1}{2}K\omega_l^2 - \frac{1}{3}BK\omega_l^3. \quad (12)$$

- (a) When the lattice undergoes a negative expansion (FM to PM) $\omega > 0$.

The sum of the last two terms in equation (12) is always negative, and the contributions of the lattice entropy change counteract the magnetic part.

(b) When the lattice undergoes a positive expansion (FM to PM) $\omega < 0$.

If $-\frac{2}{3}B\omega < 1$, the entropy change caused by the lattice effect counteracts the magnetic entropy change; if $-\frac{2}{3}B\omega > 1$, the entropy change caused by the lattice effect adds to the magnetic entropy change.

So it is estimated that the modulus of compression $K < 2.35 \times 10^7 \text{ N cm}^{-2}$ (the density of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ is 7.19 g cm^{-3} , assuming $\omega = 1\%$ at 2 T) which can be compared with the modulus of compression of iron ($K = 1.67 \times 10^7 \text{ N cm}^{-2}$) according to the prior discussions. The modulus of compression K and the coefficient B can be estimated from deformation data under different magnetic fields. Although these kinds of data are deficient, it has no influence on drawing the conclusion that the negative lattice expansion, accompanied by a first-order phase transition, always disfavors the MCE. It is attributed to the lattice cell's negative expansion why the magnitude of MCE is much lower than expected in the first-order magnetic phase transition. If the stress is *linear* with the lattice deformation, the magneto-elastic coupling is negative to MCE whether lattice cells expand or contract in the course of undergoing a first-order phase transition.

4. Summary

The metamagnetic transition of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ is investigated by means of dc and ac magnetic measurements. The dc external magnetic fields reach 10 T. With increasing external magnetic field, the Curie temperature is driven to higher temperature. During isothermal magnetization conditions, the jumps in magnetization in the vicinity of the phase transition are sharp at a few Kelvin above 196 K and smooth at a higher temperature. It can be expected that the phase transition will disappear in a strong enough external magnetic field. The MCE of $\text{LaFe}_{11.5}\text{Si}_{1.5}$ is large, but there are big discrepancies between the measurement results and the theoretical estimations. The possible phenomenological mechanism is discussed within the frame of thermodynamics. It is assumed that the stress should possess a *nonlinear* form in the elasticity limit. The lattice entropy change can be added to the magnetic entropy change when the lattice cell undergoes a positive expansion and some special conditions are met. In the conditions of zero field and high vacuum, the criterion is simplified as: if the lattice cell undergoes a positive expansion and $-\frac{2}{3}B\omega > 1$, the entropy change caused by lattice effect adds to the magnetic part; if the lattice cell undergoes a positive expansion and $-\frac{2}{3}B\omega < 1$ or the lattice cell undergoes a negative expansion, the entropy change caused by the lattice effect counteracts the magnetic entropy change. The lattice's negative expansion is always disadvantageous to achieving a large MCE. As for compounds of $\text{LaFe}_{11.5}\text{Si}_{1.5}$, the lattice cell's discontinuous negative expansion accompanied by the first-order phase transition leads to a magnetic entropy change being counteracted by the lattice part. This is the reason why a large discrepancy exists between theory and experiment.

Acknowledgments

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