

Phase Formation and Magnetocaloric Effect in $\text{La}(\text{Fe}_{1-x}\text{Co}_x)_{11.2}\text{Si}_{1.8}$ Compounds Prepared by Ball-milling

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Abstract: The samples of $\text{La}(\text{Fe}_{1-x}\text{Co}_x)_{11.2}\text{Si}_{1.8}$ ($x=0, 0.02, 0.04, 0.06$) were prepared by ball-milling with elemental powder and LaSi cast master alloys. After sintering at 1423 K for 30 min followed by quenching in water, an almost NaZn_{13} type single phase was obtained. The study of magnetic properties reveals that the Curie temperature increases with Co content from $x=0$ to $x=0.06$ whereas the magnetic entropy change decreases. The alloy of $\text{LaFe}_{11.2}\text{Si}_{1.8}$ has a maximum magnetic entropy change ΔS_m of 6.5 J/(kg·K) near its Curie temperature under a magnetic field of 0~1.5 T, while the maximum magnetic entropy change is about 2.1 J/(kg·K) with $x=0.06$. Furthermore, the magnetization of ball-milled samples exhibits a second-order magnetic transition, which is interesting for magnetocaloric applications.

Key words: magnetocaloric effect; $\text{La}(\text{Fe}, \text{Co}, \text{Si})_{13}$ compound; NaZn_{13} -type; ball-milling

Compared to conventional vapor compression systems, room temperature magnetic refrigeration based on the magnetocaloric effect (MCE) presents many advantages, such as high refrigeration efficiency, low noise, reliability and environment-friendly^[1-3]. The MCE is related to an adiabatic temperature change as well as an isothermal magnetic entropy change of a magnetic material upon application of a magnetic field. To achieve the magnetic refrigerants working at room temperature, the magnetic refrigerant with a wide range of temperatures covering room temperature and giant magnetocaloric effect (GMCE) is one of the important factors. $\text{LaFe}_{13-x}\text{Si}_x$ alloys with cubic NaZn_{13} type structure are commonly accepted as one of the most promising magnetic refrigerants. $\text{LaFe}_{13-x}\text{Si}_x$ compounds have some advantages, such as GMCE with lower price of raw materials and excluding deleterious elements. However, these alloys have two disadvantages in practical application. Firstly, the Curie temperature (T_C) is around 200 K of $\text{La}(\text{Fe}, \text{Si})_{13}$ alloys^[4], so they cannot directly be applied to room temperature magnetic refrigeration. They need to adjust T_C to room temperature while retaining their large magnetic entropy change. Secondly,

the NaZn_{13} -type structure is hard to obtain directly from common solidification process due to the intrinsic incompleteness of a peritectic reaction: $\gamma\text{-Fe} + \text{L} \rightarrow \text{La}(\text{Fe}, \text{Si})_{13}(\tau_{1a})$, which often results in the mixed phases of $\alpha\text{-Fe} + \text{La}(\text{Fe}, \text{Si})_{13}(\tau_{1a}) + \text{La}(\text{Fe}, \text{Si})_{13}(\tau_4)$ ^[5]. Thus, the traditional preparing method obviously wastes a large amount of energy and time, and restricts the application of these materials in magnetic refrigeration. Recently several researches on the synthesis of $\text{LaFe}_{13-x}\text{Si}_x$ alloys by melt-spinning have been reported^[4-9]. Shorter heat treatment is required for melt-spinning alloys compared to bulk alloys. It is the aim of this paper to examine the influence of ball-milling on the magnetic properties and the annealing time of $\text{La}(\text{Fe}_{1-x}\text{Co}_x)_{11.2}\text{Si}_{1.8}$ ($x=0, 0.02, 0.04, 0.06$) alloys. The phase relation, Curie temperature and magnetocaloric effects of $\text{La}(\text{Fe}_{1-x}\text{Co}_x)_{11.2}\text{Si}_{1.8}$ compounds are investigated.

1 Experiment

The $\text{La}(\text{Fe}_{1-x}\text{Co}_x)_{11.2}\text{Si}_{1.8}$ ($x=0, 0.02, 0.04, 0.06$) compounds were synthesized by ball-milling. LaSi alloy melt congruently at 1898 K according to Ref. [10] as precursor to prevent

Received date: November 11, 2015

Foundation item: National Natural Science Foundation of China (51161017); Scientific Research Foundation for Advanced Talents Inner Mongolia University (135145)

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oxidation of lanthanum was prepared by arc-melting under purified argon atmosphere. The LaSi ingot was ground to powder in an agate mortar. All powders including LaSi, Si and Fe were mixed in an agate mortar, and then loaded into a stainless steel vial under high-purity Ar atmosphere inside a glove box. The purities of the raw materials were larger than 99.5 wt%. Before sealing the vial, stainless steel balls were added. The powder to ball mass ratio was 1:4. The ball-milling was performed in a planetary mill (Fritsch Pulverisette-6). The as-milled powders were compacted and sealed under argon in a silica tube and annealed at 1423 K for 30 min followed by quenching in water. The phase purity and crystal structures were determined by powder X-ray diffraction (XRD) using Cu K α radiation ($\lambda = 0.154\ 184\ \text{nm}$). Magnetic measurements were performed using a vibrating-sample magnetometer (Lakeshore 7410).

2 Results and Discussion

Fig.1 shows the X-ray diffraction (XRD) pattern of LaSi alloy at room-temperature. Besides the main phase LaSi, a small amount of LaSi₂ is observed in the as-cast LaSi compound. The phase composition and structural characterization of La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} compounds from XRD are listed in Table 1. The high energy ball-milling brings about the formation of an amorphous phase in which a large amount of α -(Fe, Si) (Fig.2) appear. A short annealing treatment of 30 min at 1423 K is enough to reduce the α -(Fe, Si) content as impurity phase and obtain a well phase with cubic NaZn₁₃ type structure in all La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} alloys. According to the XRD data refinement, the main diffraction peaks of the cubic NaZn₁₃-type structure phase obviously shifts to high angle with increasing the content of Co from $x=0$ to $x=0.06$. This is a sign of lattice contraction. It attributes to the radius of Co (0.167 nm) being smaller than that of Fe (0.172 nm)^[11]. The lattice parameter gradually decreases from 1.1392 nm to 1.1388 nm with increasing Co content from $x=0$ to 0.06.

Fig.3 displays the magnetization isotherms of La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} ($x=0, 0.02, 0.04, 0.06$) compounds measured under a magnetic field of 0.05 T during the heating process. All the measurements were performed on the annealed samples. The

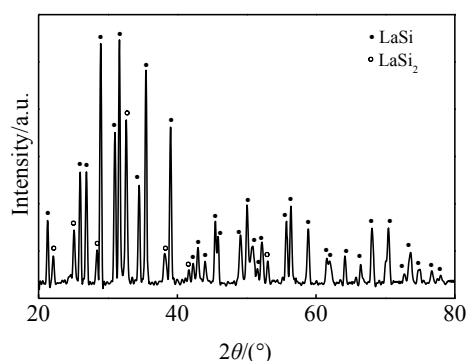


Fig.1 XRD pattern of LaSi alloy

Table 1 Structural characterization of La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} ($x=0, 0.02, 0.04, 0.06$) from XRD

x	Phases	Lattice constant, a/nm
0	NaZn ₁₃ , α -(Fe,Si)	1.1392
0.02	NaZn ₁₃ , La ₂ O ₃ , α -(Fe,Si)	1.1391
0.04	NaZn ₁₃ , α -(Fe,Si)	1.1389
0.06	NaZn ₁₃ , La ₂ O ₃	1.1388

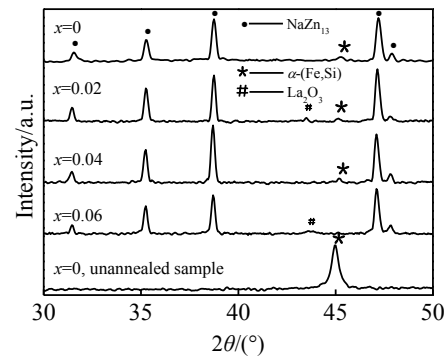


Fig.2 XRD patterns of La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} ($x=0, 0.02, 0.04, 0.06$) annealed and unannealed

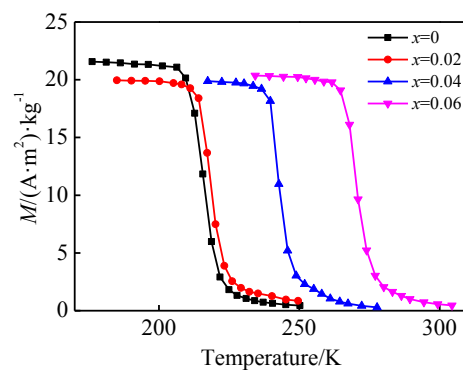


Fig.3 Temperature dependence of magnetization for La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} ($x=0, 0.02, 0.04, 0.06$) measured under a field of 0.05 T

transition temperature is determined as the maximum of dM/dT in heating process. The Curie temperature is determined to be 214.5, 217.5, 243.5, and 269.5 K for samples $x=0, 0.02, 0.04, 0.06$, respectively. The T_C of LaFe_{11.2}Si_{1.8} alloys is 214.5 K in our work, which is in accord with Ref. [11]. As previous reports^[12], the increase of Curie temperature attributes to the strong Co-Fe exchange interaction in Co-substituted La(Fe,Si)₁₃ alloys. We note the Curie temperature of the LaCo₁₃ ferromagnetic alloy, $T_C = 1297\ \text{K}$ ^[13], is much higher than that of La(Fe,Si)₁₃ alloys. It indicates that Co-Co interactions are much stronger than those of the Fe-Fe in NaZn₁₃ alloys. Thus the increase of the Curie temperature in the Co-substituted alloys of La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} should mainly attribute to the Fe-Co and Co-Co interactions.

Fig.4 displays the magnetization isotherms as a function of magnetic field of the La(Fe_{1-x}Co_x)_{11.2}Si_{1.8} ($x=0, 0.02, 0.04, 0.06$) compounds for various temperatures in the field increasing

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