



Balancing negative and positive thermal expansion effect in dual-phase La(Fe,Si)₁₃/α-Fe in-situ composite with improved compressive strength

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ARTICLE INFO

Article history:

Received 23 March 2018
Received in revised form
23 June 2018
Accepted 30 July 2018
Available online 31 July 2018

Keywords:

La-Fe-Si alloys
Magnetic phase transition
Near-zero thermal expansion
Second-order transition
Compressive strength

ABSTRACT

Materials with near-zero thermal expansion are helpful in improving the reliability of devices. One way to synthesize such materials is mixing material showing positive thermal expansion with that displaying negative thermal expansion. Large negative thermal expansion has been widely observed in NaZn₁₃-type La(Fe,Si)₁₃-based alloys during the magnetic transition. It suggests that these materials have potential applications as thermal-expansion compensators. However, how to compensate the negative thermal expansion and simultaneously improve its strength are two considerable questions. Here, we introduce in-situ precipitated α-Fe phase to compensate the negative thermal expansion of 1:13 phase, and thus achieve a near-zero thermal expansion in La(Fe,Si)₁₃/α-Fe in-situ composite. On the other hand, the distributed ductile α-Fe phase greatly enhances the compressive strength, solving the brittleness problem of 1:13 phase. The yield compressive stress of the in-situ composite with near-zero thermal expansion reaches 970 MPa. This work opens up an effective way to the design of materials with both near-zero thermal expansion and improved strength.

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1. Introduction

Most materials exhibit a positive coefficient of thermal expansion (CTE), which leads to the expanded lattices with the temperature increasing, due to the population of higher energy levels of anharmonic lattice vibrations. However, a small amount of materials contract upon heating, and this phenomenon is called negative thermal expansion (NTE). Up to the present time, NTE has been observed in the well-known ZrW₂O₈ family of materials [1–3], MnCoGe-based alloys [4,5], PbTiO₃-based compounds [6,7], ScF₃-based compounds [8], (Bi,La)NiO₃ [9], antiperovskite manganese nitrides [10–12] as well as Cellular solids with bi-materials ribs, which exhibit tunable CTE [13–24]. NTE materials show the significant application potential since they could be mixed with the materials showing positive thermal expansion (PTE) to form the composites with near-zero thermal expansion (NZTE), which is

helpful in improving the reliability of devices in numerous industrial fields [10].

In recent years, the cubic NaZn₁₃-type (1:13 phase) La(Fe,Si)₁₃-based compounds have been developed as potential NTE materials [25–28]. The observed NTE is generated from the pronounced magnetovolume effect as the magnetic ordering forms [25–28]. Large NTE has been reported in LaFe_{10.6}Si_{2.4}, LaFe_{10.6}Si_{2.4} hydrides, LaFe_{11.5}Si_{1.5}, LaFe_{11.5}Si_{1.5} hydrides, LaFe_{11.3}Co_{0.2}Si_{1.5}, LaFe_{10.5}Co_{1.0}Si_{1.5} and so on [25–27]. It suggests that La(Fe,Si)₁₃-based compounds can be used as thermal-expansion compensators to synthesize composite with NZTE during the magnetic transition. Nevertheless, such composites have been rarely reported. On the other hand, the 1:13 phase is typically brittle. As the cyclic temperature change is applied, the stress generated from large NTE (or the magnetovolume effect) tends to destroy the highly brittle parent phase and cause fractures [29–31]. This drawback greatly reduces the stability of the NTE. Hence, how to realize NZTE in La(Fe,Si)₁₃ system with improved strength is a considerable challenge.

It is known that companying the main 1:13 phase, one or two additional phases, such as α-Fe, LaFeSi or La₅Si₃, commonly appear

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in off-stoichiometric $\text{La}(\text{Fe,Si})_{13}$ alloys [32]. Among these additional phases, α -Fe is a non-rare-earth ductile phase with PTE [33]. Suppose that the prepared alloy is only composed of 1:13 and α -Fe phases, the NZTE of 1:13 phase can be offset by the PTE of α -Fe phase, so that the NZTE can be obtained in this $\text{La}(\text{Fe,Si})_{13}/\alpha$ -Fe in-situ composite by adjusting the volume ratio of the two phases. As reported by Shao et al., adding extra Fe not only increases the volume ratio of α -Fe phase, but also prevents the formation of brittle La-rich phase [34]. In this regard, $\text{La}(\text{Fe,Si})_{13}/\alpha$ -Fe in-situ composite can be naturally synthesized by adding extra Fe, and the volume ratio of the two phases can be controlled by altering Fe-content in the ingredient. Furthermore, as a toughening phase, the existence of ductile α -Fe phase blocks the crack propagation, thus leading to enhanced strength [35]. In this work, we design new compositions with more Fe addition than that reported by Shao et al., so that the volume ratio of α -Fe phase increases obviously. NZTE as well as improved compressive strength is obtained in the case when the volume ratio of 1:13 and α -Fe phases reaches 2.22:1. Additionally, we add Co element in the ingredient in order to guarantee the NTE (or the magnetovolume effect during magnetic transition) in 1:13 phase to be generated from the second-order magnetic transition, which is helpful to enlarge the temperature range in which NZTE or NTE takes place.

2. Experiments

Samples with the ingredients of $\text{LaFe}_{10.2+x}\text{Co}_{1.2}\text{Si}_{1.6}$ ($x = 0, 4, 6, 8, 10$) were prepared by arc-melting under a high-purity argon atmosphere. An excess 5 at.% of La over the stoichiometric composition was added to compensate for the loss during melting. Button ingots were melted for three times, and they were turned over each time during melting to ensure homogeneity. The as-cast samples were sealed into vacuumed quartz tubes and then annealed at 1323 K for 3 weeks before quenching into water. The reason for us to choose such a long-time annealing is to ensure the formation of 1:13 phase [36]. The room-temperature powder X-Ray diffraction (XRD) measurements were performed on a Bruker Advance D8 diffractometer. The surface of the samples was carefully polished then investigated using a FEI Quanta 250F scanning electron microscope (SEM) and optical microscope. The elemental mapping images were observed using energy-dispersive spectroscopy (EDS) in SEM. The linear thermal expansion data vs. temperature curves were obtained on NETZSCH F3 thermal mechanical analyser with a heating/cooling rate of 3 K/min using cuboid sample with a size of $3 \times 3 \times 6 \text{ mm}^3$. The reference temperature is 300 K. Magnetic properties were measured using a Quantum Design Dynacool physical property measurement system (PPMS) with a vibrating sample magnetometer. During the magnetic measurement, bulk samples with mass of $\sim 10 \text{ mg}$ were used. The heating/cooling rate is set to be 3 K/min and the magnetic field heating/cooling rate is 0.01 T/s. Compressive stress-strain measurement was carried out on a SANS-CTM5105 universal material testing machine with a strain rate of 0.0015 mm/s using the cuboid sample with a size of $3 \times 3 \times 6 \text{ mm}^3$. The volume fraction of constituent phases was

quantitatively estimated based on the SEM images by the statistics of area and the corresponding results are listed in Table 1.

3. Results and discussions

XRD patterns (Fig. 1) of $\text{LaFe}_{10.2+x}\text{Co}_{1.2}\text{Si}_{1.6}$ ($x = 0, 4, 6, 8, 10$) alloys show that our samples consist of 1:13 and α -Fe phases. With the increase of Fe content, the α -Fe volume ratio increases gradually from 12.05% in $x = 0$ –39.28% in $x = 10$ (see Table 1). Since Fe atoms have a very limited solid solubility in the lattice of 1:13 phase, the extra Fe atoms form α -Fe phase gradually [34]. On the other hand, extra Fe atoms have the potential to prevent the formation of La-rich phases [34], thus a third phase, such as LaFeSi or La_5Si_3 , is not found in Fe-rich samples. To confirm the element and phase distributions, the microstructure is observed using SEM. As shown in Fig. 2, in the SEM image of $x = 0$, a small amount of granular α -Fe grains distribute among the 1:13 phase. With the increase of x , α -Fe grains grow up and connect with each other to form dendritic-like morphology. According to the EDS analysis, it can be found that with the increase of x , both the Fe concentration in α -Fe phase and the Fe/Si ratio in 1:13 phase gradually increase (see Table 1, the Fe concentration in α -Fe phase increases from 76.41% in $x = 0$ –91.26% in $x = 10$; the Fe/Si ratio in 1:13 phase increases from 5.35:1 in $x = 0$ to 9.30:1 in $x = 10$). It can also be found that Co is distributed in both phases, but the concentration decreases with x increasing (Co concentration in α -Fe phase decreases from 9.33% in $x = 0$ –5.51% in $x = 10$; and it decreases from 8.41% in $x = 0$ –6.07% in $x = 10$ in 1:13 phase). The change of composition in 1:13 phase will obviously influence the corresponding Curie-temperature (T_C). Additionally, a small amount of Si element is detected in α -Fe phase as well. The detailed EDS results are listed in Table 1.

Due to the fact that the NTE in 1:13 phase is generated from the magnetovolume effect as the magnetic ordering forms, it is

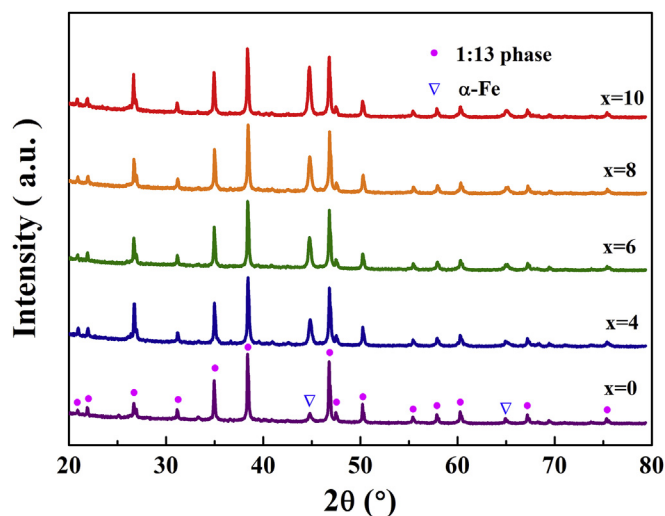


Fig. 1. XRD patterns for samples with $x = 0, 4, 6, 8$ and 10 at room temperature.

Table 1
Compositions and the corresponding volume fraction of 1:13 matrix and α -Fe phases, magnetic transition temperature (T_C), average CTE, temperature range and yield compressive stress for $\text{LaFe}_{10.2+x}\text{Co}_{1.2}\text{Si}_{1.6}$ ($x = 0, 4, 6, 8, 10$) samples.

| Sample | 1:13 phase (vol%) | α -Fe phase (vol%) | T_C (K) | Average CTE (10^{-6} K^{-1}) | Temperature range (K) | Yield compressive stress (MPa) |
|----------|--|--|-----------|--|-----------------------|--------------------------------|
| $x = 0$ | $\text{La}_{8.18}\text{Fe}_{70.28}\text{Co}_{8.41}\text{Si}_{13.13}$ (87.95) | $\text{Fe}_{76.41}\text{Co}_{9.33}\text{Si}_{14.26}$ (12.05) | 328 | – | – | – |
| $x = 4$ | $\text{La}_{7.36}\text{Fe}_{72.05}\text{Co}_{8.08}\text{Si}_{12.51}$ (77.44) | $\text{Fe}_{88.37}\text{Co}_{8.03}\text{Si}_{3.59}$ (22.56) | 307 | –9.54 | 282–304 | 864 |
| $x = 6$ | $\text{La}_{7.04}\text{Fe}_{73.53}\text{Co}_{7.36}\text{Si}_{12.07}$ (75.28) | $\text{Fe}_{89.72}\text{Co}_{6.89}\text{Si}_{3.38}$ (24.72) | 301 | –6.25 | 273–292 | 887 |
| $x = 8$ | $\text{La}_{6.87}\text{Fe}_{74.08}\text{Co}_{7.01}\text{Si}_{11.04}$ (68.98) | $\text{Fe}_{90.55}\text{Co}_{6.24}\text{Si}_{3.21}$ (31.08) | 291 | –0.655 | 261–282 | 970 |
| $x = 10$ | $\text{La}_{6.81}\text{Fe}_{78.66}\text{Co}_{6.07}\text{Si}_{8.46}$ (60.72) | $\text{Fe}_{91.26}\text{Co}_{5.51}\text{Si}_{3.22}$ (39.28) | 287 | 6.76 | 245–276 | 1004 |

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