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Spinodal decomposition in Fe–25Cr–12Co–1Si alloy under a 100 kOe magnetic field

X.Y. Sun[a], C.Y. Xu[a,b], L. Zhen[a,*], L.X. Lu[a], L.-C. Qin[b,c]

[a]School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China
[b]Department of Physics and Astronomy, University of North Carolina at Chapel Hill, Chapel Hill, NC 27599-3255, USA
[c]Curriculum in Applied and Materials Sciences, University of North Carolina at Chapel Hill, Chapel Hill, NC 27599-3255, USA

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Abstract

Spinodal decomposition in Fe–25Cr–12Co–1Si alloy subjected to thermo-magnetic treatment under a 100 kOe magnetic field was investigated by transmission electron microscopy (TEM) and Mössbauer spectrometry. The high magnetic field was found to accelerate spinodal decomposition in the early stage, but restrain the coarsening process, which led to the formation of very fine modulated structures with small aspect ratio for the ferromagnetic α_1 phase. The abnormal deterioration of magnetic properties was mainly attributed to the decrease of shape anisotropy of ferromagnetic α_1 phase for the step-aged Fe–25Cr–12Co–1Si alloy.

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Keywords: Spinodal decomposition; Magnetic field; Fe–Cr–Co alloy; Thermo-magnetic treatment

1. Introduction

Thermo-magnetic treatment (TMT) is crucial in the determination of permanent magnetic properties for Fe–Cr–Co and Alnico alloys. These two kinds of alloys undergo spinodal decomposition to form two phases structure, one is ferromagnetic and the other is paramagnetic [1–5]. External magnetic field is usually employed during isothermal treatment to elongate the ferromagnetic α_1 phase, and thus impart magnetic anisotropy to these kinds of alloys [6–8]. Generally, increasing external magnetic field intensity is expected to improve the alignment of ferromagnetic α_1 phase and increase the aspect ratio of ferromagnetic α_1 phase, which are corresponding to better magnetic properties. Chin et al. [6] performed thermo-magnetic treatment with external magnetic field up to 5 kOe on Fe–28Cr–12Co alloy. They found that the magnetic properties reached saturation when the intensity of external magnetic field (H_{ext}) exceeded 3 kOe. However, in our previous report, we observed the abnormal decline of magnetic properties for Fe–25Cr–12Co–1Si alloy when the H_{ext} intensity exceeded 8 kOe [9].

In the present work, we employed a 100 kOe magnetic field in order to verify the abnormal trends previously reported. Modulated structures at as-TMT and step aged states were characterized with transmission electron microscopy (TEM). Spinodal decomposition under a magnetic field up to 100 kOe was studied by means of Mössbauer spectrometry.

2. Experimental

The low-cobalt-type Fe–25Cr–12Co–1Si alloy (wt%) in this study was melted in a vacuum induction furnace. The ingot was homogenized at 1200 °C and then hot forged to Ø60 mm bar. Flake-like samples of $18 \times 10 \times 2.8\,mm^3$ cut from the bar were solid solution treated at 1050 °C for 1 h, and then quenched into iced brine. The same optimal parameters of isothermal ageing at 647 °C for 1 h were

*Corresponding author. Tel.: +86 451 8641 2133;
fax: +86 451 8641 3922.
E-mail address: zhenl@hit.edu.cn (L. Zhen).

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adopted for the alloy with an external magnetic field of 100 kOe. A complex step ageing process was subsequently conducted following the isothermal treatment. The detail of step ageing process was described in our previous report [9].

TEM thin foils were jet-polished and then observed on a Philips Tecnai 20 transmission electron microscope at 200 kV. Mössbauer spectrums were obtained at room temperature in standard transmission geometry with a conventional constant acceleration spectrometer using a ^{57}Co source in a Pd matrix. Magnetic properties were measured with a DC automatic hysteresisgraph (Model NIM-200) at room temperature.

3. Results

3.1. Modulated structure

It is generally believed that increasing external magnetic field intensity can elongate the ferromagnetic α_1 phase during thermo-magnetic treatment, and thus introduce shape anisotropy for Fe–Cr–Co alloys that undergo spinodal decomposition within the miscibility gap. For example, a 2 kOe magnetic field produced ferromagnetic α_1 phase with aspect ratio of 3.6, and an 8 kOe magnetic field gave aspect ratio of 4.7 for as-TMT Fe–25Cr–12Co–1Si alloy, as shown in Fig. 1(a) [9]. However, in the present study, very fine two-phase microstructure was observed for the as-TMT alloy subjected to thermo-magnetic treatment with a 100 kOe magnetic field, as shown in Fig. 1(b). The average diameter of ferromagnetic α_1 phase is about 15 nm and the aspect ratio is about 2. The aspect ratio is even much smaller than that treated with low intensity of 2 kOe.

3.2. Magnetic properties

The magnetic properties of Fe–25Cr–12Co–1Si alloy as a function of external magnetic field intensity were shown in Fig. 2. As illustrated in our previous report [9], B_r, ${}_iH_c$ and $(BH)_{max}$ all increased with the increase of H_{ext} intensity,

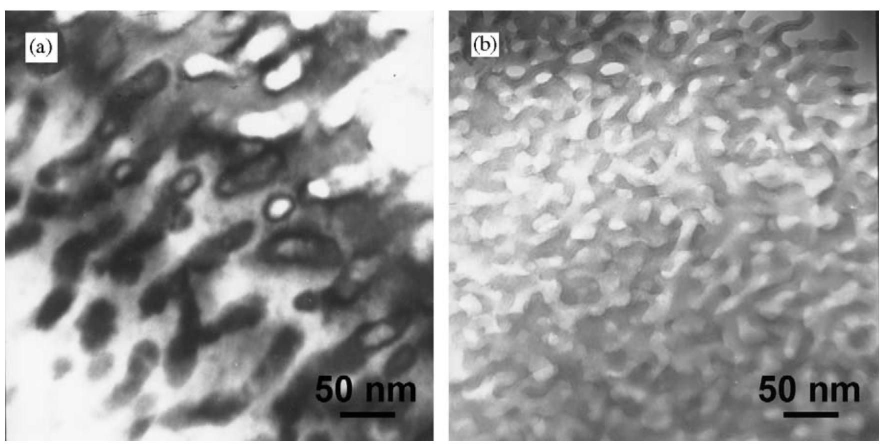

Fig. 1. TEM micrographs of the as-TMT Fe–25Cr–12Co–1Si alloy subjected to thermo-magnetic treatment with different H_{ext} intensities: (a) 8 kOe and (b) 100 kOe.

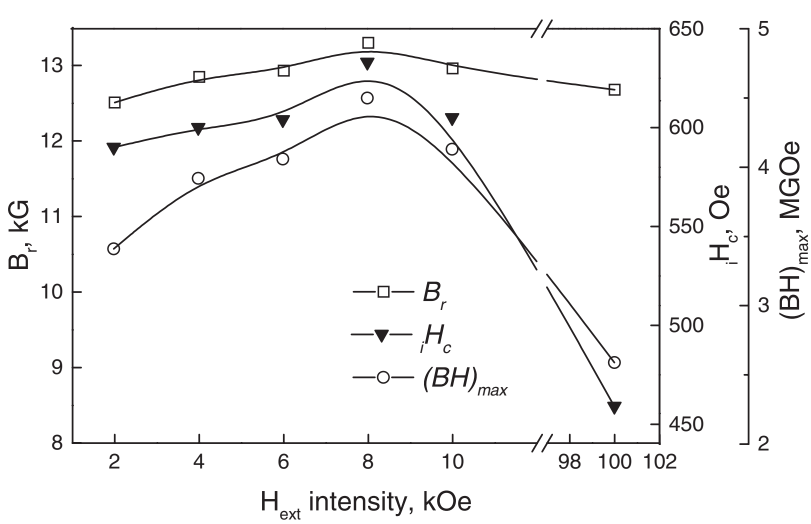

Fig. 2. Variation of magnetic properties with intensity of external magnetic field for the step-aged Fe–25Cr–12Co–1Si alloy.

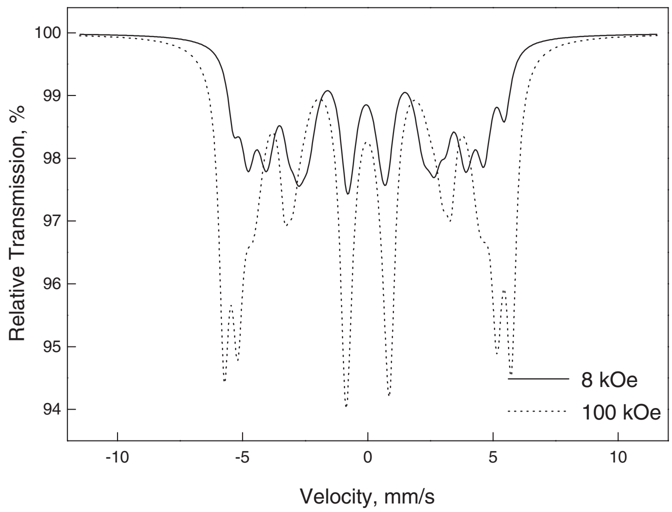

Fig. 3. Mössbauer spectrums of the as-TMT Fe–25Cr–12Co–1Si alloy subjected to thermo-magnetic treatment with different H_{ext} intensities.

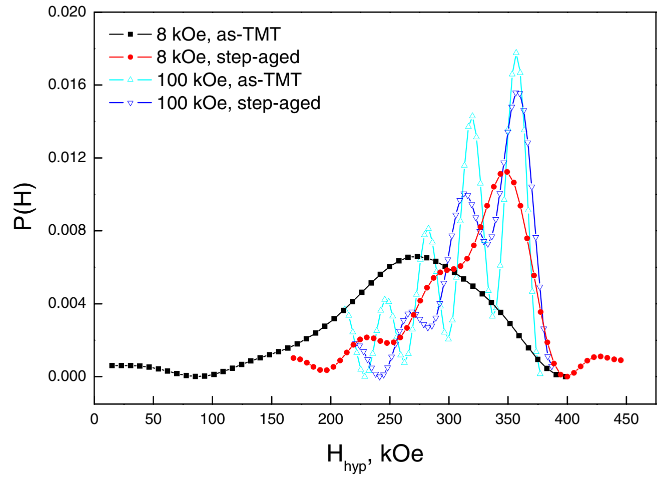

Fig. 4. Hyperfine field distribution of the as-TMT and step-aged Fe–25Cr–12Co–1Si alloy subjected to thermo-magnetic treatment with different H_{ext} intensities.

and reached the maximum values at an 8 kOe magnetic filed. Then, B_r, ${}_iH_c$ and $(BH)_{max}$ all declined slightly when the intensity of applied magnetic field increased from 8 to 10 kOe. In the present work, thermo-magnetic treatment in a 100 kOe magnetic field was performed with the same alloy in order to verify the abnormal decline tendency of permanent magnetic properties. The same optimal thermo-magnetic treatment parameters were adopted here. It was found that the permanent magnetic properties did decrease further when thermo-magnetic treatment was carried out in 100 kOe magnetic field, compared with that treated with 10 kOe magnetic fields (Fig. 2). The results obtained were consistent with the previous reported abnormal decline tendency of magnetic properties when H_{ext} intensity exceeds a critical value, which is 8 kOe here for Fe–25Cr–12Co–1Si alloy.

4. Discussion

Fig. 3 gives the room temperature Mössbauer spectrums of the as-TMT Fe–25Cr–12Co–1Si alloy thermo-magnetically treated with 8 and 100 kOe magnetic field, respectively. Typical sextets were found for both spectrums, indicating the superposition of several subspectrums. The enhancement of relative transmission for each sextet with 100 kOe external magnetic field indicates that larger composition differentia between the two phases of α_1 and α_2, which is due to the acceleration of spinodal decomposition induced by high magnetic field, especially at early stage. The acceleration of spinodal decomposition was further confirmed by the distribution of hyperfine field $P(H)$, as shown in Fig. 4. The $P(H)$ profile of the as-TMT alloy treated with 8 kOe magnetic field roughly follows Gauss distribution, indicating a wide variation of iron environment due to the presence of Cr and Co atom. The $P(H)$ profile shifts towards high field region and is distributed in a narrower region when thermo-magnetic treatment was performed under a 100 kOe magnetic field. More peaks at high magnetic field region suggest the decrease of Cr atoms around Fe and Co atom, which suggests the acceleration of spinodal decomposition during thermo-magnetic treatment. The average hyperfine field deduced from $P(H)$ profile for the as-TMT Fe–Cr–Co alloy with 100 kOe magnetic field is 318.0 kOe, which is much higher than that of 256.9 kOe in the case of 8 kOe magnetic field, as shown in Table 1. This further suggests the acceleration of spinodal decomposition during thermo-magnetic treatment. The difference of Vickers hardness for the as-TMT alloy due to different H_{ext} intensities offers further demonstration. The composition difference between modulated two phases increases with the further proceeding of spinodal decomposition. Thus the mismatch degree becomes larger, which results in the increase of Vickers hardness, as shown in Table 2.

Table 1
Average hyperfine field of Fe–25Cr–12Co–1Si alloy thermo-magnetically treated with different intensities of external magnetic field

H_{ext} intensity	Average hyperfine field (kOe)	
	As-TMT	Step aged
8 kOe	256.9	327.8
100 kOe	318.0	331.9

Table 2
Vickers hardness of Fe–25Cr–12Co–1Si alloy thermo-magnetically treated with different intensities of external magnetic field

H_{ext} intensity	HV (kgf/mm^2)	
	As-TMT	Step aged
8 kOe	264.0	415.2
100 kOe	319.8	397.8

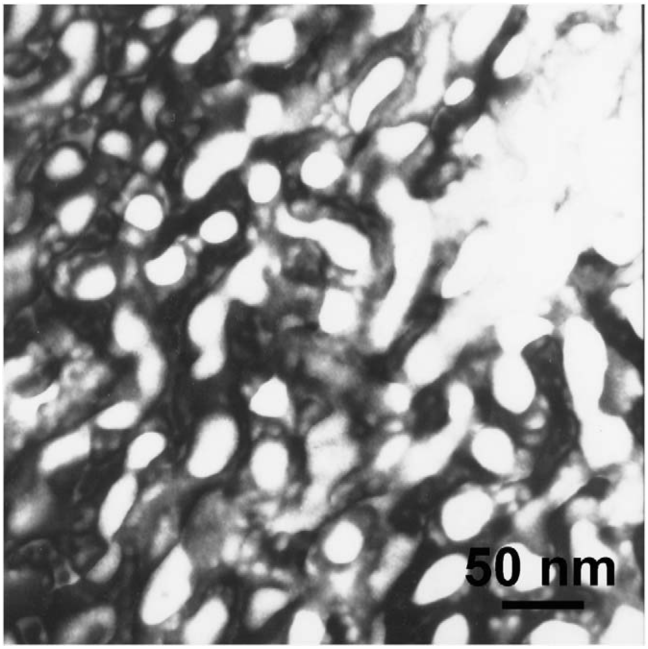

Fig. 5. TEM micrograph of the step-aged Fe–25Cr–12Co–1Si alloy thermo-magnetically treated with H_{ext} intensity of 100 kOe.

The effect of magnetic field on the free energy curve cannot be ignored if H_{ext} is high enough, and the chemical driving force of precipitated ferromagnetic phase is enhanced, which leads to the increase of decomposing speed at early stage. On the other hand, once modulated structures are formed, there exists phase interface between precipitated phase and the matrix. The potential barrier of atoms diffusing increases with the increase of H_{ext} intensity. In the case of 100 kOe magnetic field, the atom diffusing across phase interface might be blocked if the potential barrier is high enough, resulting in the frozen of coarsening process during spinodal decomposition [10]. Thus very fine modulated structures would be formed under high magnetic field, as shown in Fig. 1(b).

After step-ageing, the $P(H)$ files are of the similar shape but broader distribution is observed in the case of 8 kOe magnetic field, which indicates wider variation of iron environment. In the case of 100 kOe magnetic field, the peak for the step-aged alloy shifts slightly towards high magnetic field region compared with that if as-TMT alloy. The increase of interspacing between two peaks indicates further decomposition at lower temperatures during step ageing. The average hyperfine field of the step-aged alloy subjected to thermo-magnetic treatment with 100 kOe magnetic field is 331.9 kOe, which is just a little higher than that of 327.8 kOe for the case of 8 kOe magnetic field. As for Vickers hardness, the difference is also very small after step ageing. The lower HV value in the case of 100 kOe might be attributed to the secondary decomposition at a low temperature, as shown in Fig. 5.

Since no external magnetic field is applied during step ageing, the modulated structure undergoes coarsening at lower temperatures. Fig. 5 gives TEM micrograph of the step-aged alloy. The step-aged alloy treated with 100 kOe magnetic field is seems to exhibit no shape anisotropy since the ferromagnetic α_1 phase are almost spherical. Thus, a comparison of coercivity between the step-aged Fe–Cr–Co alloy thermo-magnetically treated with different H_{ext} intensities is made here. Approximate 31% of the coercivity for the step-aged alloy with 8 kOe magnetic field can be attributed to the shape anisotropy of ferromagnetic α_1 phase as a result. The contribution of shape anisotropy is comparable to the calculated value of 28% [11], indicating that the decline of magnetic properties was mainly due to the loss of shape anisotropy of ferromagnetic α_1 phase.

5. Conclusions

Abnormal deterioration of permanent magnetic properties was observed for Fe–25–12Co–1Si alloy subjected to thermo-magnetic treatment in a 100 kOe magnetic field, compared with that treated with an optimal magnetic field with relatively low intensity of 8 kOe. Mössbauer spectrum and hardness measurement results indicate the acceleration of spinodal decomposition under high magnetic field. The high magnetic field is believed to restrain the coarsening process, resulting in very fine modulated structures. The decline of magnetic properties is mainly attributed to the decrease of shape anisotropy of ferromagnetic α_1 phase.

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