Microstructure and Magnetostriction of Rapid-Solidified Fe-15 at% Ga Alloy*1

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Melt-spun, rapid solidified Fe-Ga ribbon sample exhibited large magnetostriction and good ductility as compared with conventional bulk sample. But the origin was not clear yet. In order to investigate the occurrence of large magnetostriction in Fe-Ga ribbon sample, the correlation between magnetostriction and the crystal grain morphology was inspected in detail by SEM/EBSP method for Fe-15 at% Ga alloy. In comparison with as-spun ribbon sample, short-time (0.5 h) heat treated ribbon had stronger [100] oriented texture and exhibited larger magnetostriction of 140 ppm (×10−5) at 800 kA/m. These phenomena suggest that such a large magnetostriction is caused by the release of considerable large internal stresses in as-spun ribbon as well as the remained strong textures after annealing.

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1. Introduction

Magnetostrictive materials are ideally required to have high strength, good ductility, large magnetostriction at low saturation magnetic field, high magnetomechanical coupling coefficients and low cost for engineering applications. Intermetallic compound, Terfenol-D single crystal exhibits giant magnetostriction $\varepsilon = 2000$ ppm at room temperature. However, Terfenol-D single crystal is expensive and brittle to be worked.

In recent years, Clark et al. studied magnetic, magnetostrictive and elastic properties of Fe$_{100-x}$Ga$_x$ (15 $\leq$ $x$ $\leq$ 20) single crystals, which exhibited large magnetostriction of 300 ppm in low magnetic field, durability and ductility.1–3) Fe$_{100-x}$Ga$_x$ (17 $\leq$ $x$ $\leq$ 23) single crystals which were water-quenched from temperatures $\geq$800°C4,5) with body-centered cubic structure exhibited larger magnetostriction.4,5)

In our previous study,5) we developed Fe-Ga rapidly solidified ribbons by melt-spinning method so as to overcome the technical problem in single crystal. The ribbon sample showed large magnetostriction and good ductility as compared with the conventional melt-worked bulk sample with randomly oriented crystal grains.

In this study, in order to investigate the occurrence of large magnetostriction of rapid-solidified Fe-Ga ribbon samples, we applied heat treatment the ribbon samples to obtain larger magnetostriction of rapid-solidified Fe-Ga ribbon samples, and then the ribbon under loading stress $\sigma$ of 10 MPa was measured by the strain gauge method. Since the ribbon sample was thin foil, the ribbon was weighted on the bottom of the sample. The grain boundary character distribution (GBCD) was measured by electron backscatter diffraction pattern (EBSP) from the surface of the ribbon sample. The grain boundary character distribution (GBCD) defined by the orientation difference between the adjacent crystals i.e. mutual orientation relationship was inspected. X-ray diffraction (XRD) profiles from the surfaces were obtained using CuK$_{\alpha}$ radiation at room temperature for each sample.

Crystal orientation was defined by orientation imaging microscopy (OIM), which analyzed electron backscatter diffraction pattern (EBSP) from the surface of the ribbon sample. The grain boundary character distribution (GBCD) defined by the orientation difference between the adjacent crystals i.e. mutual orientation relationship was inspected. X-ray diffraction (XRD) profiles from the surfaces were obtained using CuK$_{\alpha}$ radiation at room temperature for each sample.

Magnetiization $M$ was measured by vibrating sample magnetometer (VSM) method. Magnetostriction $\varepsilon$ was measured by the strain gauge method. Since the ribbon sample was thin foil, the ribbon was weighted on the bottom and then the ribbon under loading stress $\sigma$ of 10 MPa was hanged in the magnetic field center as shown in Fig. 1. The ribbon samples were fixed by being attached on upper and lower aluminum plates with glue, where the lower plate could move only along vertical direction with weight. Moreover, magnetostriction was determined by averaging values obtained from strain gauges on both surfaces.

2. Experimental Procedures

Ingot of Fe-15 at% Ga was prepared from electrolysis iron (99.999% pure) and gallium (99.999% pure) by arc melting. It was homogenized at 1173 K for 24 h. The ribbon samples with $80 \mu$m thickness and $5 \text{ mm}$ width were produced from the ingot by single-roll melt-spinning method in argon atmosphere. In order to investigate the effect of heat treatment on magnetostriction, the ribbon samples (12 mm in long, 5 mm in width, $80 \mu$m in thickness) were annealed for 0, 0.5, 6.0 h at 1173 K in a vacuum atmosphere and subsequently quenched into icy water.

In order to investigate the texture of ribbons in detail, the inverse pole figure and the grain size for Fe-15 at%Ga were prepared by being attached on upper and lower aluminum plates with glue, where the lower plate could move only along vertical direction with weight. Moreover, magnetostriction was determined by averaging values obtained from strain gauges on both surfaces.

3. Results and Discussion

3.1 X-Ray diffraction pattern

X-ray diffraction patterns of three Fe-15 at% Ga ribbons (as-spin, annealed for 0.5 and 6.0 h at 1173 K) are shown in Fig. 2. Three ribbon samples have the body-centered cubic structure with the lattice constant $a = 0.2897$ nm. The intensity of (200) peak for the 0.5 h ribbon sample is stronger than other peaks. The result suggests that the ribbon has [100] oriented texture. Moreover, the tendency is enhanced by short-time heat treatment.

3.2 Pole figure

In order to investigate the texture of ribbons in detail, the inverse pole figure and the grain size for Fe-15 at% Ga were

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analyzed by SEM-EBSP/OIM system.

Figure 3 shows inverse pole figure color code maps of (a) as-spun and (b) annealed for 6.0 h Fe-15 at%Ga ribbons. The red color exhibits the axis of grain orients to crystalline [001] direction. Figure 3(c) also is photograph of cross section observed by laser microscope for as-spun ribbon. The columnar microstructure is parallel to the normal direction to the plate.

The [001] pole figures for three ribbons annealed for 0 h, 0.5 h and 6.0 h at 1173 K are shown in Fig. 4, where RD and TD mean directions parallel and perpendicular to the rolling direction of ribbon (see Fig. 1). The ribbons short-annealed for (a) 0 h and (b) 0.5 h clearly show sharp texture. It is seen that the [001] axes of many grains in Fig. 4(a) orient in the
3.3 Grain size distribution

Figure 5 shows grain size distribution for three annealed ribbon samples (0 h, 0.5 h and 6.0 h) analyzed by OIM based on SEM/EBSP method where the vertical and the horizontal axes show appearance frequency and grain size, respectively. The average grain size of the 0 h and the 0.5 h ribbon samples is in the range of 6.3 to 7.9 μm. This result suggests that the grain size does not change drastically by short time heat treatment. On the contrary, the average grain size of the 6.0 h ribbon increases to 10 μm. As shown in Fig. 3(b), the polycrystal consists of both the fine and coarse grains after recrystallization.

3.4 Distribution of grain boundary misorientations and grain boundary character

Figure 6 shows distribution of grain boundary misorientations in three ribbons. The vertical and horizontal axes show appearance frequency and misorientation angle between grains, respectively. Most of the misorientation angles for 0 h and 0.5 h ribbons are distributed from 15° to 45° and from 15° to 30°, respectively. On the other hand, those for 6.0 h ribbon are distributed from 35° to 55°, that is in the range of high angles. These results are consistent with the distribution of [001] poles.

Next, we researched an effect of heat treatment for Fe-15 at%Ga ribbon to grain boundary character distribution (GBCD), that was crystal arrangement. Figure 7 shows the frequency of coincidence boundaries as a function of Σ for three ribbons, where Σ is reciprocal density of common sites in Coincidence Site Lattice (CSL). As shown in Fig. 7, the ratio of Σ1 (relative rotation angle between adjacent grains <15°) is 21.6%, 32.1% and 25.8% for the 0 h, 0.5 h and 6.0 h ribbon samples, respectively. These results indicate that the rapidly solidified Fe-15 at%Ga ribbon mostly consists of low angle grain boundaries of which energy is low and has characteristic texture. The ratio of Σ3 increases in the 6.0 h ribbon sample. The Σ3 is coherent twin boundary formed by recrystallization. The ratio of random grain boundaries is about 60% in these three ribbons.
3.5 Magnetization and magnetostriction

The ribbon samples could be rotated around the RD. Magnetic field was applied perpendicular to RD and the strain was measured with increasing \( \theta \) from 0 to 90°, where \( \theta \) was the rotation angle between the transverse direction (TD) of ribbon and applied magnetic field (See Fig. 1).

Figure 8 shows the \( M-H \) loops of as-spun Fe-15 at%Ga ribbon. Magnetization for \( \theta = 0° \) is quickly saturated in a weak magnetic field less than 160 kA/m and its saturation magnetization is \( 230 \times 10^{-6} \) Wb·m·kg\(^{-1} \) which is 83.6% value of pure iron. On the other hand, for \( \theta = 80° \), because of occurrence of strong demagnetizing field, there is no sign that magnetization is saturated even after the field of 800 kA/m is applied. \( M-H \) loops of other two ribbon samples exhibit similar behavior.

Next, we investigated dependency of magnetostriction on \( \theta \) for as-spun Fe-15 at%Ga ribbon, which had an easy axis of magnetization, [001], near \( \theta = 80° \) direction. Figure 9 shows magnetostriction \( \varepsilon \) for \( \theta = 0°, 40°, 60° \) and 80° as a function of \( H \). The \( \varepsilon \) was estimated by averaging values obtained by
two gauges put on both faces of sample under loading stress \( \sigma = 10 \text{ MPa} \) with no bending. Longitudinal magnetostriction \( \varepsilon \) (TD at \( \theta = 0^\circ \)) reaches easily a saturation value of 30 ppm in rather weak applied field of 160 kA/m. Other \( \varepsilon \) for \( \theta = 40^\circ, 60^\circ \) and \( 80^\circ \) increase continuously with \( H \). Among these, a maximum strain is obtained for \( \theta = 80^\circ \), and \( \varepsilon \) reaches 90 ppm at \( H = 800 \text{kA/m} \). It can be judged from these results that the large magnetostriction originates in the strong [001]-oriented texture near \( \theta = 80^\circ \).

Figure 10 shows dependency of magnetostriction of Fe-15.1 at\%Ga ribbon on heat-treatment for (a) TD and (b) RD at \( \theta = 80^\circ \). For 1173 K-6.0 h ribbon, \( \varepsilon \) of TD is approximately saturated to reach 60 ppm at \( H = 640 \text{kA/m} \). On the other hand, for 1173 K-0.5 h and as-spun ribbons, \( \varepsilon \) of TD increases with \( H \), reaching about 140 ppm and 90 ppm at \( H = 800 \text{kA/m} \), respectively. In the case of RD, \( \varepsilon \) for three ribbons increases with \( H \) in similar fashion and there is no or small difference between them. From these results, it is found that \( \varepsilon \) of the ribbon increases with strengthening [001]-oriented texture and has small hysteresis.

It is considered that this phenomenon is caused by the unique microstructure of the ribbon which has the strong [001]-oriented texture possessing fine columnar grains with low angle grain boundaries, where magnetic domain rotation and magnetic wall movement are not restrained so much.
4. Conclusions

In this study, we have investigated the correlation between the magnetostriction and the crystal grain morphology of Fe-15 at%Ga ribbons produced by melt-spinning method in order to reconfirm the occurrence of large magnetostriction. The crystal grain morphology has been measured by SEM/EBSP and XRD. Magnetic properties have been also measured. The main conclusions are as follows:

1) As-spun ribbon has the strong [001]-oriented texture possessing fine columnar grains, where the [001] axis tends to be oriented in the range of 0° to 40° from pole in the pole figure. This tendency becomes stronger by short annealing the ribbon.

2) Rapidly solidified ribbon consists of grains with many low-angle grain boundaries where magnetic domain rotation and magnetic wall movement are not restrained so much.

3) Largest magnetostriction occurs in short heat-treated Fe-15 at%Ga ribbon. That results from both released internal stresses and strong orientation to [001].

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REFERENCES


