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Neutron diffraction studies of magnetostrictive Fe–Ga alloy ribbons

Xuegen Zhao, a) Nigel Mellors, Susan Kilcoyne, and Don Lord
Institute for Materials Research, University of Salford, Salford M5 4WT, United Kingdom

Nicoleta Lupu and Horia Chiriac
National Institute of Research and Development for Technical Physics, Iasi 700050, Romania

Paul F. Henry
Institut Laue-Langevin, F-38042 Grenoble, France

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Melt-spun Fe–Ga ribbons were prepared and some ribbons were annealed at 1000 °C for 1 h then slowly cooled to room temperature. X-ray diffraction patterns revealed no evidence of texture and only bcc phase in the as-quenched ribbons. However, high-resolution neutron diffraction patterns gave more information on the structure of these ribbons. Only diffractions from the disordered bcc A2 phase were found in as-quenched ribbons with 15, 17.5, and 19.5 at. % Ga content, without any trace of satellite peaks or splitting peaks from the proposed Ga–Ga pairing superlattice structure. The broadening of the base of the (110) peaks for all samples except the as-quenched 15 at. % Ga ribbon might indicate the existence of some kind of short range ordering. Ribbons developed L12 phase after annealing especially in the Fe 19.5 at. % Ga ribbon where the formation of L12 phase reduced the Ga content in the remaining A2 phase and decreased its lattice parameter dramatically. D03 phase formed in the as-quenched 22.5 at. % Ga ribbon and the following annealing treatment transformed more A2 phase into D03 phase. © 2008 American Institute of Physics.

I. INTRODUCTION

The Fe–Ga system is characterized by a large solid solubility of Ga in bcc α-Fe, 11 at. % at room temperature, and up to 36 at. % at 1037 °C. 1 However, appreciable amounts of Ga well in excess of the solubility limit can be retained in the metastable disordered bcc solid solution phase (A2 phase) at room temperature by rapid cooling. 1–3 The replacement of Fe by Ga results in a large increase in magnetostriction, approaching 400 ppm for quenched single crystals 4–6 at room temperature, with low saturation fields of several hundreds of oersted, and over −2000 ppm has been reported for melt-spun Fe–Ga ribbons. 7,8 The magnetostriction λ100 maximizes around 17–19 at. % of Ga and then decreases with increased Ga content. 9 The decrease of λ100 at higher Ga content coincides with the change of structure from the disordered bcc to a low-energy long range ordered phase (D03). Local short-range ordering of the Ga atoms along the [100] direction in the disordered Fe structure has been suggested to be responsible for this increase10 but experimental confirmation still remains a challenge. 11,12 This is because the peaks corresponding to both D03 and A2 phases overlap except for the weak satellite peaks corresponding to the ordered structure. These satellite peaks can generally not be clearly identified due to the very low relative intensities found using x-ray diffraction. Neutron diffraction is expected to give better results as the difference in atomic scattering factor between Fe and Ga atoms is bigger in neutron diffraction and the atomic scattering factor does not change with the changing scanning angle. 13

II. EXPERIMENT

Ingots of Fe100−xGa x (x=15, 17.5, 19.5, 21, and 22.5) were prepared by arc melting high purity (>99.9) constituent elements multiple times to ensure homogeneity. High vapor pressure of Ga and the use of care in the melting procedures resulted in negligible Ga loss. Ribbon samples were produced by melting 3 g of ingot with an induction coil in a partial argon atmosphere and ejecting the melt onto a rotating copper wheel at a wheel speed of 35 m/s. The orifice diameter of the quartz crucible was approximately 0.5 mm. Isothermal heat treatment was performed on some ribbons at 1000 °C for 1 h followed by slow cooling to room temperature. Ribbons were evacuated in a quartz tube and then sealed under approximately 1 atm of argon before heat treatment.

X-ray diffraction examinations were performed using a Bruker D8 Advance diffractometer. The θ-2θ scans were obtained using Cu Kα radiation in a Bragg–Brentano configuration. The x-ray diffraction patterns obtained were used to determine the phases present and the crystallographic texture of the samples. Neutron diffraction experiments were carried out on both as-quenched and annealed Fe100−xGa x (x=15, 17.5, 19.5, and 22.5) alloy ribbons on the D2B diffractometer, ILL at
FIG. 1. Neutron diffraction patterns of as-quenched and annealed Fe–Ga ribbons.

Grenoble, France. Ribbon samples were packed into vanadium cans and scanned from 0° to 158° at ambient temperature by using the high-resolution mode with a scanning step size of 0.05°.

III. RESULTS AND DISCUSSION

The x-ray diffraction patterns of as-quenched melt-spun ribbons show no evidence of obvious texture. In addition to the primary reflections at 2θ~44° (110) and ~64° (200), no reflections corresponding to the DO₃ phase were observed.

The neutron diffraction patterns from both melt-spun ribbons and annealed ribbons are shown in Fig. 1. The six strong diffraction peaks can be easily identified as arising from (110), (200), (211), (220), (310), and (222) reflections from the bcc (A₂ phase) structure or correspondingly (220), (400), (422), (620), and (444) reflections from the DO₃ structure. The ratios of peaks compared with the strongest diffraction peak [(110) for A₂ phase or (220) for DO₃ phase] intensities are almost identical among samples.

However, after enlarging the low intensity part of the diffraction patterns, as shown in Fig. 2, we can see some weak diffraction peaks, in addition to the six strong peaks. One group of them belongs to the vanadium container used in the experiment. Vanadium, being bcc with a = 3.0297 Å, has diffraction peaks close to the left of A₂ peaks and are clearly shown in the diffraction pattern of Fe 15 at. % Ga melt-spin ribbon. When the Ga content is increased to 22.5 at. %, extra peaks from the ordered Fe₃Ga DO₃ structure are observed.

It is mentioned in several papers that Ga–Ga pairs formed along the (100) direction may be responsible for the high magnetostriction found in Fe–Ga alloys. This kind of ordering results in a structure change from bcc to tetragonal and should give some satellite peaks or even peak splitting. These peaks are not expected to be identified from the DO₃ phase as they will overlap. However, we cannot see any satellite peaks from such a superlattice structure or a noticeable splitting of the observed peaks for ribbons with 15, 17.5, and 19.5 at. % Ga, although these ribbons show only disordered A₂ phase. Broadening of the base of the (110) peak for all samples except the as-quenched 15 at. % Ga ribbon might indicate the existence of some kind of short range ordering, although the possibility of thermal diffuse scattering from phonons could not be excluded considering that the dramatic elastic softening occurs in these materials with Ga doping.

More peaks were observed in the patterns from samples which had been annealed at 1000 °C for 1 h then slowly cooled down to room temperature, especially with x = 19.5 at. % Ga. These new peaks have been indexed as coming from a Fe₃Ga L₁₂ phase. It was a surprise to observe this phase appearing in these annealed ribbon samples as it is believed that the L₁₂ phase is very difficult to form and we have not seen such a phase in annealed Fe–Ga bulk samples. However, this annealing treatment has not developed a significant volume of the DO₃ phase in the 19.5 at. % Ga ribbon and no obvious Bragg peaks from DO₃ can be seen even in the low intensity part of the diffraction pattern. This may indicate a structural transformation from A₂ to L₁₂, rather than from DO₃ to L₁₂ during the annealing process.

It is difficult to tell how much A₂ phase exists in the 22.5 at. % Ga ribbons. No obvious peak splitting can be seen with the peaks above 60°, in fact, they can be easily fitted with a single-peak profile. However, the percentage of the
It can be seen from Fig. 4 that the ribbon sample with 19.5 at. % Ga shows a significant change in lattice parameter after annealing treatment. The as-quenched ribbon has A2 phase with \( a = 2.9045 \ \text{Å} \). After heat treatment, the ribbon has developed ordered \( L1_2 \) \( Fe_3Ga \) phase and possibly \( D_0_3 \) \( Fe_3Ga \) phase as well that reduce the Ga content in the A2 phase and, thus, the lattice parameter reduces down to 2.9014 Å. Using this parameter and the relation between composition and lattice parameter obtained from the annealed ribbons with 15 and 17.5 at. % Ga, it is estimated that the annealed 19.5 at. % ribbon might have an actual Ga content of 18.1 at. % for the A2 phase, and the volume fraction of \( L1_2 \) phase could be around 20%.

Both \( D_0_3 \) and \( L1_2 \) \( Fe_3Ga \) phases have different magnetic properties to disordered A2 \( Fe–Ga \) alloys, and will therefore act as impurities in the A2 matrix, such as rare-earth inclusions in Terfenol-D, decreasing the magnetostrictive performance of the materials. If element (M) substitution or the addition method, in addition to quenching, can be used to hinder the formation of second phase material such as \( D_0_3 \), while still maintaining a high Ga content and high Curie temperature, then it may be possible to obtain much higher magnetostriction from \( Fe–Ga–M \) alloys.

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