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Relation between order degree, damping behavior and magnetic response in Fe-Si and Fe-Al-Si alloys

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Neutron diffraction, mechanical spectroscopy and magnetic loops tests were performed in ordered Fe-based alloys. It was found that independently of the type of order, $D0_3$ or B2, the mobility of dislocations and grain boundaries is markedly reduced in ordered alloys. In contrast, when the order decreases or disappears after annealing, the dislocation and grain boundary mobility increases. The magnetic response of ordered alloys has been found dependent on the order degree, but also of the defects arrangement in the sample promoted by thermal treatment.

Introduction

Despite the large use in technological applications of iron-silicon and iron-aluminium-silicon alloys [1,2], there are still some basic questions that must be studied in further detail as for instance, the correlation between the thermo mechanical state and the magnetic response. Therefore, the aim of this work is to show a correlation between the order degree and the defects configuration in these alloys and their effects on the magnetic response. Neutron diffraction (ND), mechanical spectroscopy (MS) and magnetic hysteresis loops tests were used as experimental techniques in the present work.

Experimental

Samples

Samples of composition (at.%): Fe-10Si, Fe–12Al–12Si and Fe-6Al-9Si were used in this work. Samples were homogenised at 1323K during 1 hour under high vacuum, followed by quenching into room temperature (RT) water [3,4].

Measurements

ND studies were performed at D20 and D1B powder diffractometers in the Institute Laue Langevin (ILL), Grenoble, France, using neutron wavelengths of λ = 1.3Å and λ = 2.52 Å, respectively. Diffractograms were obtained under high vacuum in situ during heating. MS measurements were performed on heating and cooling runs, so called thermal cycles, under high vacuum. Magnetic hysteresis loops were measured at RT and plotted curves are the average over ten measurements.

Results

Figure 1 shows the (100) peak corresponding to the B2 superlattice reflection for a Fe-10Si alloy both in the as-quenched state and after slow cooling down in the fur-

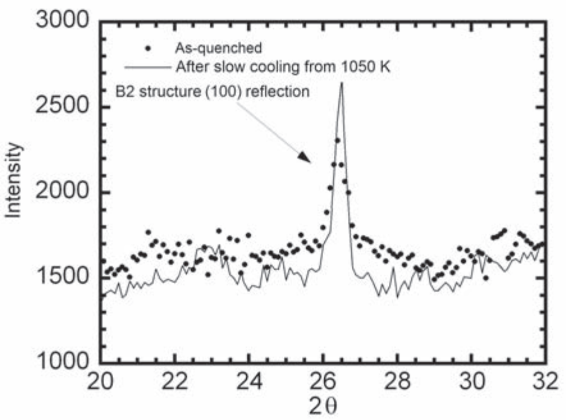

Figure 1. B2 superlattice (100) neutron diffraction reflection corresponding to the as-quenched state (filled circles) and after slow cooling in the furnace (solid line).

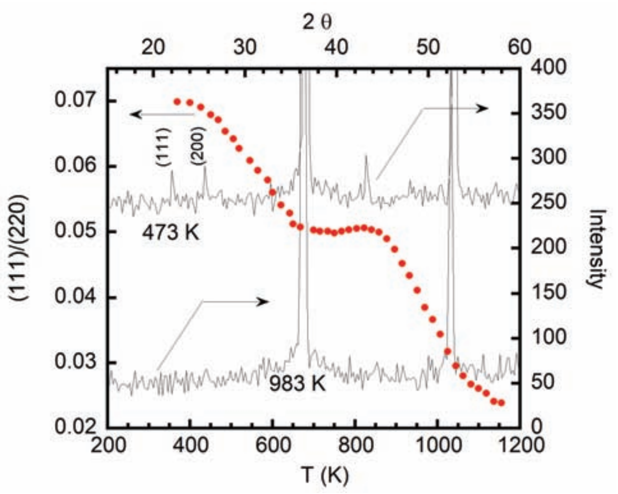

Figure 2. Lower and left axis: Evolution of the relative integrated intensity of the (1 1 1)/(2 2 0) reflection as a function of temperature for a Fe-12Al-12Si sample. Upper and right axis: Evolution of the $D0_3$ order temperature for a Fe-6Al-9Si sample.

nace. As shown, a small increase in the integrated area after thermal treatment can be observed indicating that the order in the as-quenched state is not complete. Then, a further increase in the measuring temperature gives rise to the final ordering process to B2 [3].

Fe-12Al-12Si sample exhibited at RT the appearance of $D0_3$ order [4]. Figure 2 shows the evolution of the relative integrated intensity of the (1 1 1)/(2 2 0) reflection as a function of temperature for a Fe-12Al-12Si sample. As it can be seen from the figure, the degree of order decreases as the temperature is increased. In contrast, during the cooling of the sample the order degree is restored [4].

Fe-6Al-9Si sample exhibited at RT, the appearance of $D0_3$ order, Figure 2. Above 983K the order disappears, as shown by the disappearance of the (111) and (200) reflections related to the $D0_3$ structure. In addition, during the cooling, after a previous heating up to 1150K, the order is restored approximately at the same temperature (983K) [4].

Figure 3 shows the damping (Q^{-1}) spectra for a Fe-10Si sample after successive thermal cycles. All spectra show a characteristic grain boundary relaxation peak (GB), P1, around 800 K [3]. As it can be seen from the figure, the peak temperature of the maximum related to P1 moves towards higher temperatures during the cycles up to 973 K. In the second heating the shift is very small, being larger during the third run up in temperature. In contrast, when the sample was previously measured up to 1050K, the peak temperature of the maximum during the heating run is shifted towards smaller temperatures (full line). Besides this, the peak temperature is very close to the initial peak temperature corresponding to the as-quenched sample. Nevertheless, the damping spectrum changes strongly when the sample was thermally treated up to 1273K [3].

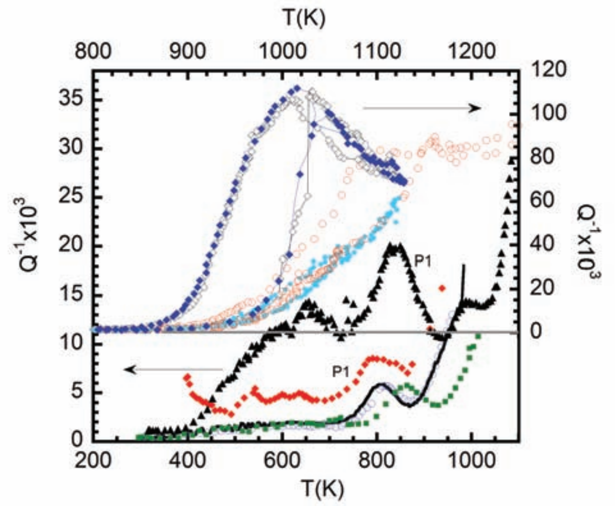

Figure 3. Lower and left axis: Damping spectra for a Fe-10Si sample. Sample in the as-quenched state: full rhombuses. After a heating run up to 973K: empty circles. After two heating runs up to 973K: full squares. After a heating run up to 1050K: full line. After a heating run up to 1273K: full triangles. Upper and right axis: Damping spectra for Fe-12Al-12Si (circles) and Fe-6Al-9Si (rhombuses). Full circles: thermal cycle up to 1130K. Empty circles: thermal cycle up to 1200 K.

The damping spectra measured for 12Al-12Si sample, during thermal cycles up to two different final temperatures are also shown in Figure 3 by shifting the base-line (upper and right axis). The damping spectra increase monotonously with the temperature increase and no relaxation peaks have been found during the thermal cycles up to 1130K. The heating and cooling runs are similar with a small thermal hysteresis. However, increasing the temperature above 1200K during the heating process leads to the appearance of a damping peak during the subsequent cooling at around 1100K [4].

For Fe-6Al-9Si samples a thermal hysteresis in the damping of about 100K appears in the temperature interval 850K-1050K (rhombuses, upper and right axis in Figure 3). In addition, increasing the final temperature of the thermal cycles up to 1200K did not modify the damping behaviour [4].

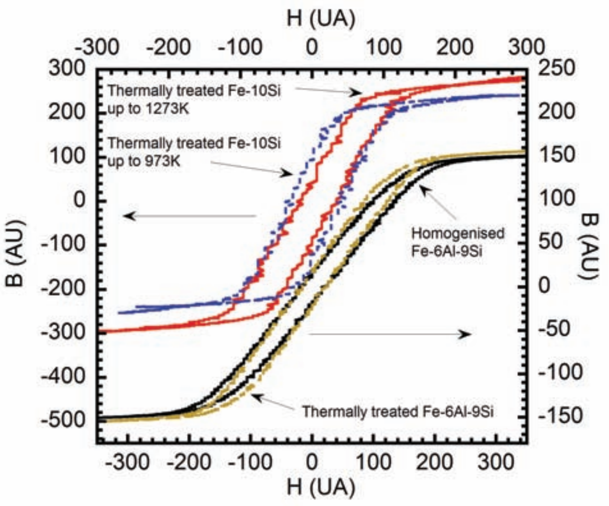

Figure 4. B-H curves. Lower and left axis: Fe-10Si sample. Upper and right axis: Fe-6Al-9Si sample.

Figure 4 shows the behavior of the induction (B) against applied magnetic field (H) hysteresis loops corresponding to Fe-10Si samples in different thermo mechanical states. Sample thermally treated up to 1273K has a magnetic loop similar to the loop corresponding to the as-quenched sample. Nevertheless, the largest difference in the magnetic response appears for the sample thermally treated up to 973K, which shows larger coercitivity [5].

The behaviour of B against H for a Fe-6Al-9Si sample in the homogenised state and after thermal treatment is also shown in Figure 4. The sample after the thermal treatment up to 1200K exhibits a larger value of the induction of saturation (20%) and a smaller coercive force (3%). However, for samples Fe-12Al-12Si clear changes in the magnetic loops after the thermal treatment cannot be found [6].

Discussion

As it was already shown from Figure 1 the as-quenched Fe-10Si alloy is not fully B2 ordered and has a lot of quenched-in-defects (Figure 3). The recovery of the quenched-in-defects occurs, during heating, at temperatures below 850 K [3]. During the slow cooling from the different maximum temperatures, the B2 order restores and the final order degree is higher than the corresponding to the as-quenched alloy. In addition, this sample has recovered partially the quench-in-defects. The increase in the order degree after reaching 973K produces an increase of internal stresses generated by the reorganization of defects in the ordered B2 phase. In particular dislocation must move in pairs towards the grain boundary where they are locked, reducing the grain boundary mobility [3]. This explains the small increase in the peak temperature of GB in the second heating run. During heating up to 1050K the sample transforms according to the phase diagram. The B2 phase transforms to a bcc at 973K and for higher temperatures the sample is disordered and the super dislocations disappear. Then, the structure can relax internal stresses, which were retained in the ordered B2 superlattice. This effect leads to shift the peak temperature of GB towards smaller temperature. Besides, a thermal treatment at temperatures above 1273K leads to a large recovery of defects in the disordered lattice, giving rise to increased damping values in a subsequent warming run [3].

The increase in the quantity of order in Fe-10Si leads to the deterioration of the magnetic properties, which is revealed by the increase in coercitivity in thermally treated samples at 973K. Nevertheless, the comparison of both thermally treated samples, which have the same order degree but different coercitivity, allows establishing that the order degree is not the single parameter controlling the magnetic behaviour [5]. Consequently, it should be highlighted that the defects must be considered at the same time that the order effects to determine the magnetic behaviour.

For Fe-12Al-12Si sample, the monotonous increase of the damping as a function of temperature without the appearance of a damping peak during the thermal cycles up to 1130K and the further appearance of the damping peak during the cooling after the heating up to 1200K, can be explained by considering the evolution of the degree of order and the defect configuration in the sample as a function of temperature. The decrease of the order degree by heating the sample above 1200K enhances the dislocations and grain boundaries mobility making easier the recovery of the as-quenched-dislocations. This leads to a rearrangement of grain boundary dislocations revealed through the appearance of the damping peak at around 1100K during the cooling after the pre-heating to 1200K [4].

The damping behaviour for a Fe-6Al-9Si sample exhibits a large hysteresis between the heating and the cooling during the thermal cycles both up to 1130K and 1200K. Indeed, Fe-6Al-9Si sample is $D0_3$ ordered at RT, Figure 2. However, at temperatures close to 983K, the $D0_3$ structure changes to bcc in agreement with the ternary phase diagram [4]. This temperature is close to the one where the damping starts to increase strongly (980K). This kind of behaviour is in agreement with the neutron diffraction results and phase diagram. After the $D0_3$→bcc transition, the order degree is reduced and then the mobility of dislocations and grain boundaries is enhanced, leading to a recovery of the microstructure. Consequently, again, the GB relaxation peak appears during the cooling process [4].

Regarding to the magnetic behaviour in Fe-6Al-9Si sample, the thermal treatment performed during the thermal cycles improve the magnetic behaviour. Nevertheless, in Fe-12Al-12Si, where the order is larger than for Fe-6Al-9Si sample, the improvement of the magnetic behaviour after the thermal treatment cannot be found [6].

Conclusions

In ordered Fe-based alloys independently of the type of order, $D0_3$ or B2, the mobility of dislocations and grain boundaries is markedly reduced. In contrast, when the order decreases or disappears after annealing, the dislocation and grain boundary mobility increases. The magnetic response of ordered alloys has been found dependent on the order degree, but also of the defects arrangement in the sample promoted by thermal treatment.

Acknowledgments

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