Mobility of dislocations and grain boundaries controlled by the order degree in iron-based alloys

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Abstract Neutron diffraction and mechanical spectroscopy studies were performed in Fe-Al-Cr, Fe-Al-Si and Fe-Si alloys. Results show that in ordered alloys independently of the type of order, D03 or B2, the mobility of dislocations and grain boundaries is markedly reduced. It is revealed through small values both of damping and of the strength of the amplitude dependent damping (S). Moreover, the behaviour of S as a function of temperature is a suitable tool for determining the order changes as a function of temperature.

1. Introduction
In ordered lattices, dislocations should move in pairs, giving rise to the so called superdislocations, which leads to a decrease in their mobility regarding to the disordered lattice. Then, the recovery of quenched-in-defects and -dislocations has a slow rate; affecting also to the grain boundary mobility. Neutron diffraction (ND) and mechanical spectroscopy (MS) studies were coupled in this work for correlating the order degree and its effect on the mobility of dislocations and grain boundaries in Fe-Al-Cr, Fe-Al-Si and Fe-Si alloys. Indeed, MS is a very sensitive technique for studying the interaction processes between defects in materials [1].

2. Experimental
Samples of two iron based ternary systems, Fe-Al-Cr and Fe-Al-Si, and binary Fe-Si have been studied in this work. Sample composition, in at.% , are: Fe-25Al-8Cr, Fe-25Al-25Cr, Fe-12Al-12Si and Fe-10Si; respectively labelled as: TCr8, TCr25, TSi12 and B10. Sample details are described in Ref. [2-4] for ternary systems and in Ref. [5] for Fe-Si binary samples.

ND studies on ternary alloys were performed at the D1B powder diffractometer in the Institute Laue Langevin (ILL), Grenoble, France. The employed neutron wavelengths for Fe-Al-Cr and Fe-Al-Si alloys were \( \lambda = 2.52\text{Å} \) and \( 1.28\text{Å} \), respectively. For Fe-Si alloys ND studies were performed at the D20...
installation at the ILL, using a neutron wavelength of $\lambda = 1.3\,\text{Å}$. In situ measurements, in both diffractometers, were performed during heating at 3K/min from room temperature up to different maximum temperatures under vacuum ($10^{-2}\,\text{Pa}$).

MS, measurements were performed in an inverted torsion pendulum at frequencies close to 1 Hz under vacuum ($10^{-5}\,\text{Pa}$). Damping and square frequency, were measured with an error less than 2%. The measurements were carried out during subsequent heating and cooling runs, both at 1K/min, on the same specimen. Amplitude dependent damping (ADD), i.e. damping as a function of the maximum strain on the sample, $\varepsilon_0$, was calculated by evaluating the derivative of the curve corresponding to the decaying areas of the torsional vibrations as a function of the period number by means of Chi-square fitting. The strength of ADD effects is measured through $S$ parameter ($S = \frac{\Delta Q^{-1}}{\Delta \varepsilon_0}$).

3. Results and Discussions

Figures 1.a and 1.b show a three-dimensional picture (intensity, $2\theta$ and temperature) of the phase evolution “in situ” observed at the diffractometer for samples TCr8 and TCr25, respectively. On heating, the TCr8 sample exhibits two consecutive order→disorder transitions: the first one a $\text{D0}_3 \leftrightarrow \text{B2}$ transition at 833 K from a cubic structure (space group $\text{Fm} \overline{3} \text{m}$) ordered at the next-nearest neighbours to a cubic one ordered at nearest neighbours (space group $\text{Pm} \overline{3} \text{m}$) and a second disordering process at 973 K to a $\text{I} \overline{m} \text{3} \text{m}$ cubic structure, $\text{D0}_3 \leftrightarrow \text{B2}$ transition. On the other hand, the TCr25 sample only exhibits the $\text{B2} \leftrightarrow \text{bcc}$ transition at 973 K.

![Figure 1](https://example.com/figure1.png)

**Figure 1**: “In situ” neutron thermo-diffraction patterns measured during heating for as-quenched samples, (a) (TCr8) Fe-25Al-8Cr and (b) (TCr25) Fe-25Al-25Cr.

ND patterns at room temperature after the first “in situ” heating show differences in the degree of order between this annealing state and the as quenched one. For instance Fig. 2 shows the room temperature spectra for binary B10 sample, both in the as-quenched state and after annealing up to 1170 K. A small increase in the integrated area after thermal treatment above 1050 K can be observed indicating that the order in the as-quenched sample is not complete. No changes in the full width at half maximum of the diffraction peaks were found for all the group of studied samples, which indicate that changes in the grain size do not occur during heating; in agreement with the metallurgical observations [2, 3].
Figure 2. Neutron diffraction patterns for binary B10 sample at room temperature, as quenched state (continuous line), after the first heating (dots) and at 1170 K (dashed line).

The damping measured at increasing amplitudes is at first a constant, then increases sharply above amplitudes of around $10^{-6}$ [1, 6]. This behaviour has been explained by Granato and Lücke as due to the breaking away of dislocations loops from pinning impurities [7]. Under increasing applied stress, the lengths of dislocations between pinning points bow out until the dislocation escapes from pinning points. This mechanism of break-away of dislocation from weak pinning points give rise to the appearance of amplitude dependent damping (ADD). Nevertheless, ADD can be also used for studying heavy plastically worked materials by considering the average free length of dislocations [8]. Therefore, the order changes occurring in alloys as a function of temperature should be described by means of ADD since the decrease in the dislocation mobility in the ordered lattice. It leads to smaller bow outs and then smaller amount of breaking away processes due to the smaller swept areas by the dislocation movement. Indeed Figure 3 shows the behaviour of the S parameter as a function of temperature for the TCr8 sample, during the first heating from 600 K up to 1050 K. In addition, the evolution of the squared root of the intensities for the (200) and (111) reflections, which are proportional to the order parameter ($\eta$) for the D0$_3$ and B2 structures respectively, is also shown in Figure 3. S exhibits a slope change at the temperatures where the order state is modified. The first increase in S appears for the D0$_3$ → B2 transformation at 833K and the second one appears for the B2 → bcc transition at 973 K. In fact, dislocations should move in pairs in the ordered lattice, which leads to a decrease in their mobility. A decrease in the order degree enhances the mobility of the dislocation line, allowing sweep larger areas by bowing. It leads to a larger amount of weak pinning points inside the swept area, where the dislocation could escape during its movement, giving rise to the increase in S values. The sharp S increase above 973 K is promoted by the increase in the dislocation mobility in the disordered phase, which is in agreement with the transition B2 → bcc. Besides, the peak-shaped in S at around 890 K can be related to the change in mobility for dislocations between the transition D0$_3$ → B2.

Similarly to the behavior exhibited by sample TCr8, sample B10 shows a marked increase in S at the temperature where the (200) peak intensity begin dropping and the B2 → bcc transition develops (973 K), due to the increase in the dislocation mobility in the disordered phase, Figure 4. In addition, TSi12 sample show an increase in S for temperatures where the D0$_3$ order start to decrease, i.e. over around 860 K, Figure 4. Then, regardless of the sequence of order-disorder transitions observed the reduction of long-range order degree implies an increase of the damping linked to a higher mobility of dislocations.
Figure 3: $S$ as a function of temperature and square roots of intensities of the diffraction peaks for a type TCr8 sample. Vertical arrows indicate the transitions temperatures.

Figure 4: $S$ (lower and left axis) and square root of intensities of the diffraction peaks (upper and right axis) for: TSi12 (circles) and B10 (triangles).

Figure 5: Lower and left axis: Damping spectra for a B10 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 TSi12 (circles). Full circles: thermal cycle up to 1130K. Empty circles: thermal cycle up to 1200 K.
In the other hand, Figure 5 shows the damping spectra for a B10 sample after successive thermal
cycles performed “in situ” at the spectrometer. All spectra show a characteristic grain boundary
relaxation peak, P1, around 800 K [5, 9]. 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 fact, 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 (see Figure 2), leading to an increase in the internal stresses [5, 9]. 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). Indeed, during the 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 superdislocations disappear. Then, the structure
can relax internal stresses which were retained in the ordered B2 superlattice. Nevertheless, the damping
spectrum changes strongly when the sample was thermally treated up to 1273 K (full triangles, Figure
3), due to a large recovery of defects in the disordered lattice. The damping behaviour after successive
heating ramps depends strongly on the temperature limit achieved. Heating below the order-disorder
temperature (973 K) restores the order degree increasing internal stresses. In the case of passing the
disordering temperature the structure of the dislocations change drastically enhancing the relaxation of
stresses.

The damping spectra measured for the TSi12 sample, during thermal cycles up to two different final
temperatures are also shown in Figure 5 by shifting the base-line. The damping spectra increase
monotonously with the temperature increase and no relaxation peaks have been found during thermal
cycles up to 1130 K. The heating and cooling runs are similar with a small thermal hysteresis. However,
by increasing the temperature above 1200 K during the heating process, it leads to the appearance of a
damping peak during the subsequent cooling at around 1100 K [2, 9]. In addition, for TCr8 and TCr25,
the damping behavior as a function of the thermal cycles resulted similar to the exhibited by sample
TSi12, but the hysteretic behaviour develops after thermal cycles up to around 1050 K [3]. In fact, the
appearance of the hysteretic behaviour on damping is controlled by the decrease in the order degree,
which leads to an increase in the mobility of dislocations and grain boundaries, giving rise to the
appearance of the grain boundary peak related to the solute during the cooling run [2, 3, 9].

Therefore, as it can be inferred from the above results, the behaviour of S as a function of
temperature follows the changes in the order state during the warming of the sample, i.e. the
modification of the order state is viewed at the temperature where it occurs. In contrast, usual damping
measurements as a function of temperature (Figure 5) could reveal the appearance of order changes after
successive heating and cooling runs up to temperatures over the order-disorder temperature transition.

4. Conclusions
In ordered alloys independently of the type of order, D03 or B2, the mobility of dislocations and grain
boundaries is markedly reduced, which is shown through small values of both damping and S. An
excellent correlation between the order behaviour and S was found. A decrease in the order degree leads
to an increase in S, due to the dislocation mobility increases. In addition, when the order decreases or
disappears after annealing, the grain boundary mobility also increases.

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