STUDY OF DISLOCATION MOBILITY IN 304 STAINLESS STEEL

L.F.C.F. Lima
(Energy and Nuclear Research Institute, IPEN, São Paulo, Brazil)
P.E.V. Miranda and S.N. Monteiro
(Federal University of Rio de Janeiro, Rio de Janeiro, Brazil)

ABSTRACT

Internal friction, IF, measurements were carried out in a type AISI 304 austenitic stainless steel, SS, at approximately 1 Hz of frequency in the temperature interval from 120 to 573K. The IF and the frequency spectra were obtained in samples which had gone through specific heat treatment. The results showed IF spectra with a well defined peak at 260K. The intensity of this peak depends on the amount of plastic deformation previously introduced in the sample. Another broad peak was detected between 300 and 400K. Both peaks could only be detected after plastic deformation whether torsion or uniaxial tension. In torsionally deformed samples at 77K the IF spectrum shows a high damping due to a possible phase transformation which occurs around room temperature, RT. The broad peak at higher temperature is sensitive to recovery induced by linear annealings.

INTRODUCTION

An understanding of the behavior of crystalline materials such as metals, during plastic deformation, can only be achieved through analysis of dislocation movements. This is due to the fact that the displacement of these defects is the main cause of plastic slip in the crystals. Among the various mechanisms which can control dislocation motion, one can mention, their interaction with obstacles, such as the crystal lattice, other dislocations or point defects. Identification of these obstacles can be made by combining specific thermomechanical treatments with the use of among others the IF technique. This permits new alloys with better mechanical strength or ductility to be designed controlling the participation of these obstacles.

The behavior of the type AISI 304 SS observed by IF has been evaluated in several conditions [1-5]. Igata et al. [1] (utilizing medium frequencies) observed three peaks after plastic deformation at RT, the intensities of which increased with the amount of deformation. These peaks were explained by the authors as being due to dislocation release from blockages caused by carbon, nitrogen and carbon/nitrogen compounds. Subsequently [2] a new peak was observed at 360K, in samples deformed at low temperature, LT, which disappears after annealing at 573K, and was attributed to the existence of α' martensite. The low frequency IF tests of Lima and Miranda [3] and Lima et al. [4], with samples deformed in tension at RT and at 523K, respectively, showed a peak at 260K with an activation energy of 0.75 eV and another at 370K which was sensitive to linear annealings. A recent paper of Quiroga et al. [5], work carried out at low and medium frequencies, after deformation at 80K, not only confirms the previous results [1], but also report a peak at 360K with the following characteristics; a) it appears only after LT deformation; b) it anneals out during the measurement; c) it is not thermally activated and d) its dependence on the amount of deformation follows perfectly that of the ϵ phase, that occurs in austenite upon LT deformation.

Inspite of the references mentioned above many questions still remain unanswered to understand the behavior of austenitic SS tested by IF. This motivated the present work in which the objective has been to investigate the evolution of the IF peaks of 304 SS deformed in torsion at liquid nitrogen and at room temperatures.

EXPERIMENTAL

Wire samples of 304 SS with 1.2 mm diameter (with the following composition in weight percent: 0.059 C, 17.8 Cr, 7.9 Ni, 0.022 P, 0.5 Si, 2.0 Mn, 0.38 Mo, Fe: balance) were encapsulated in a quartz tube under pressure of 1.33×10^{-1} Pa, heated at 1173K during 600 s and quenched in water. After this treatment the average grain size was 30 µm.

The samples were deformed in torsion at 77K or at RT.

The IF and frequency measurements were carried out in an inverted torsion pendulum at a frequency of about 1 Hz and over temperatures ranging from 120 to 573K. The free decay method was used for the measurements and the maximum surface strain amplitude was 1.3×10^{-5}. The heating rate was $0.02^{\circ}\ s^{-1}$.

RESULTS

Fig. 1 shows the IF (a) and the frequency (b) curves as a function of temperature for 304 SS cold-worked 9% in torsion at 77K. Subsequently the sample was heated to RT for mounting and then cooled to 120K to carry out measurements. Curve a shows a narrow peak at 360K. In curve b, it can be observed that there is no significant frequency change in the temperature range corresponding to the IF peak.

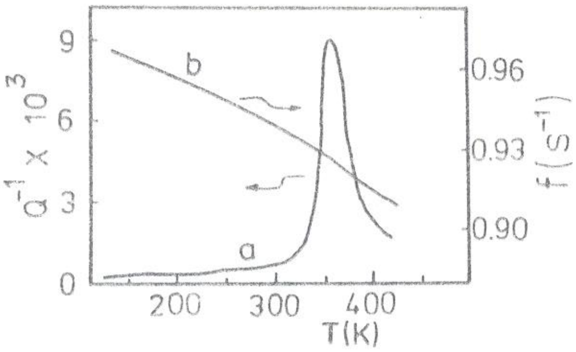

Fig. 1 IF (a) and frequency (b) curves of a sample annealed at 1173K and 9% cold-worked in torsion at 77K, after heating to RT for mounting.

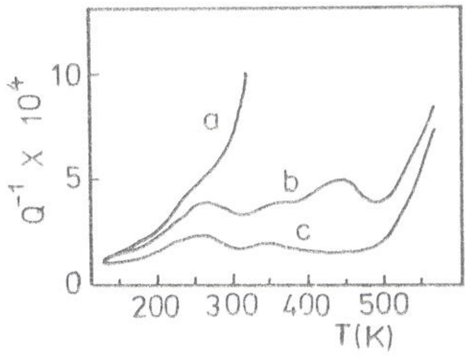

Fig. 2 IF curves of a sample annealed at 1173K and 9% cold-worked in torsion at 77K: a) after mounting, and then annealed at: b) 430K and c) 573K.

Fig. 2 shows the IF curves as a function of temperature obtained with the sample cold-worked 9% in torsion at 77K: a) after mounting; and after linear annealing at: b) 430K and c) 573K. Curve a in this figure corresponds to the LT region of curve a in Fig. 1. Comparing curve a in Fig. 1 with curve b in Fig. 2 it can be observed that linear annealing at 430K has reduced the value of the IF maximum by about a factor of twenty. On the other hand linear annealing at 573K affects the spectrum, by not only lowering the background but also by eliminating the peak at 430K.

Fig. 3 shows the IF curves as a function of temperature obtained with a sample cold-worked 9% in torsion at RT: a) after mounting; and after linear annealing at: b) 373K, c) 473K and d) 573K. These annealings at intermediate temperatures have been carried out to obtain information about changes in the IF and frequency spectra. Comparing curves a and b in Fig. 3, it can be observed that annealing at 373K causes alterations, mainly in the region above 300K. Finally, comparison of curve c in Fig.2 with curve d in Fig. 3 reveals that although the specimens were deformed at different

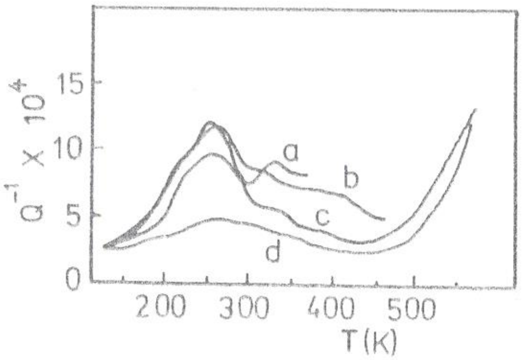

Fig. 3 IF curves of a sample annealed at 1173K and 9% cold-worked in torsion at 293K: a) after mounting, and then annealed at: b) 373K, c) 473K and d) 573K.

temperatures, their behavior in terms of IF after linear annealing at 573K are similar.

DISCUSSION

The results presented in Figs. 2 and 3 confirm those obtained with the same extent of deformation in tension at 523K (Fig. 3 of |4|) concerning the significant effect of plastic deformation. Comparing the IF curves as a function of temperature for samples deformed in torsion at 77K (Figs. 1 and 2) and at 293K (Fig. 3), and in tension at 523K (Fig. 3 of |4|) it can be observed that: a) the LT deformation results in a microstructure that contributes to an important energy absorption at 360K, although the frequency (dynamic elastic modulus) (curve b, in Fig. 1) decreases moderately, b) in curve b of Fig. 2 which corresponds to the sample deformed at 77K and then annealed at 430K, the curves of the three samples reveal well defined peaks at 260K and, a region between 300 and 480K, where at least two peaks overlap, c) the RT deformation is the more efficient in developing the 260K peak, since its height is about three times that of the corresponding peaks obtained upon deformation at 77K and 523K, d) the 77K deformation induces in the material, a microstructure that favours the formation of a peak at about 430K not observed after deformation at higher temperatures and e) the linear annealing at 573K provokes a change in the microstructure in such a way that the curves progress to a common form, characterized by a reduction in the 260K peak and the near extinction of the peaks situated above 300K.

The results reported in this paper, in |3|, |4|, by Igata's group |1,2| and by Quiroga's group |5|, make clear the complexity of the phenomena responsible for the observed curves. To facilitate analysis the curves are considered separately and a possible correlation attempted. The curves can be divided into two parts corresponding to the temperature intervals, 120 to 300K and above 300K. Moreover the 360K peak, observed after LT deformation will be considered separately.

Interval 120 to 300K - Although there are at least two peaks in this interval, Igata et al. |1| have observed three, the more important peak being situated at 260K, which appears always after cold-work and its height increases with the extent of deformation until a saturation value |1-4|. Moreover, its height and its variation in terms of the linear annealings, are functions either of temperature or the deformation mode. Hence at the moment it is difficult to add to the conclusions of some of the authors |3,5|, that the relaxation process is due to the dislocation point defects interaction in the γ phase.

Interval above 300K - In this interval there are at least two peaks and a background that increases sharply at 500K. The LT deformation is responsible for the generation of a new peak at 440K, Fig. 2, not cited in the literature. All these peaks suffer a sensible reduction when the material recovers, suggesting that this behavior is an effective microstructural rearrangement due to the linear annealing at 573 K (Figs. 2 and 3). Linear annealings at intermediate temperatures also modify the IF curves as shown in Fig. 3. In this temperature range (300 to 573K) either dislocation rearrangement or point defects migration and phase transformation can occur, making it thus difficult to analyse the whole curve, without the use of supplementary techniques.

Maximum at 360K - This peak has appeared after plastic deformation in torsion at 77K. Its width is lower than that of a Debye peak and the peak anneals out during a linear anneal at 430K, curve b in Fig. 2. This peak is therefore due to a microstructural transformation. It is well known that plastic deformation of 304 SS, at low temperatures, can induce two martensitic phases, the ϵ (hcp) and α '(ccc) phases. Their stability as a function of aging, has been studied by Mangonon and Thomas |6,7|, utilizing X-rays difractometry. It has been observed |8,9| that the ϵ phase begins to disappear with aging at 423K, which sould suggest that the reversion to the γ phase begins at this temperature. In the present case, the start of the changes takes place just after RT and, besides, in general, phase transformation is accompanied by an important variation in the elastic modulus (frequency) that has not been verified (curve b in Fig. 1). It has been observed |6,7| that the ϵ phase was hcp instead of fcc, containing a high density of stacking faults distributed at random. Thus it is possible that the deformation mode (tension) favours the first (ϵ phase) and the torsion mode the second one (α' phase). In this case, the peak observed could be related to the recombination of partials into perfect dislocations, due to the increase of stacking fault energy with temperature or simply to stress relief. However, the constriction of partial dislocations can represent the beginning of retransformation of ϵ phase in austenite.

This peak seems to correspond to the X and HT peaks observed by Igata et al. |2| and by Quiroga et al. |5|, respectively at the same temperature. Although in the present work the peak's height is higher, the deformation modes were different; Igata's peak was due to deformation in tension (extent of deformation comparable) at 77K, and Quiroga had rolled his samples at 80K. The latter, as already mentioned in the introduction verified that the peak appeared only after rolling at LT and annealed out during the measurement and that it was not thermally activated and its dependence on extent of deformation followed perfectly that of the ϵ phase, observed also by Mangonon and Thomas |6,7|. These facts lead to the conclusion that the peak is sensible to the recovery of the deformed microstructure, in which the ϵ and α' phases are present, besides the γ phase. On the other hand, Igata's group, which has also utilized X-rays difractometry, did not observe the ϵ phase, and therefore associated the peak to the α' phase.

CONCLUSION

IF measurements carried with samples of a 304 SS showed the existence of various peaks, which appear only after plastic deformation of the material. One of them at 360K, observed only after LT deformation, is not a relaxation peak and has been associated to the recovery of the deformed substructure, probably due to recombination of partials dislocations in the γ phase. Another peak is well defined at 260K (1Hz). In the region between 300 and 480K there is a complex of at least two peaks and yet one more at 430K if the deformation is at LT. The definition of the exact mechanism corresponding to each one of these peaks requires further experimental data under specific conditions.

REFERENCES

1. Igata N et al., J. de Phys., 42 (1981), C5-193.
2. Igata N, Chen HB and Miyahara K, Scripta Met., 16 (1982), 1039.
3. Lima LFCP and Miranda PEV, J. de Phys., 46 (1985), C10-135.
4. Lima LFCP et al., In: Proceedings of the 7th Brazilian Congress of Engineering and Materials Science, Florianópolis, SC, Brazil (1986), p. 257.
5. Quiroga J et al., J. de Phys., 46 (1985), C10-661.
6. Mangonon PL Jr and Thomas G, Met. Trans., 1 (1970), 1577.
7. Mangonon PL Jr and Thomas G, Met. Trans., 1 (1970), 1587.
8. Holzworth ML and Louthan MR Jr, Corrosion - NACE, 24 (1968), 110.
9. Reed RP, Acta Met., 10 (1962), 865.