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MICROPLASTICITY AND INTERNAL FRICTION IN PURE IRON

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Abstract.- We have studied the microdeformation behaviour of pure iron at low temperature (4K - 250K). Two thermally activated microdeformation stages are observed : one is associated to the α process observed by internal friction, the other is linked to the β_α peak. Activation parameters determined for the α component are in good agreement with double-kink formation on non screw dislocations. β_α component is due to dislocation depinning from an extended atmosphere.

1. Introduction.- Generally, the relaxation spectra of plastically b.c.c. metals are composed of three bands of internal friction peaks according to the Chambers nomenclature : α , β and γ [1, 2, 3, 4]. Peak α is generally attributed to double-kink nucleation on non screw dislocations, Peak β to point defect-dislocation interaction and peak γ to double kink generation on screw dislocations. Each relaxation process is characterized by specific activation energies and volumes, which can be determined by internal friction and microdeformation experiments.

In this paper, we study the microdeformation and the internal friction of a pure iron. Many experiments were performed on iron [2]. Generally a peak α is observed around 30K (1hz), with a shoulder α' at lower temperatures. The α peak is attributed by Hivert et al. [5] and by Astie et al. [8] to the kink generation on non screw dislocations. Furthermore, for low plastic deformation, a β_α peak is observed by Ritchie et al. [6] and Dufresne et al. [7], attributed to thermo-mechanical unpinning of dislocation.

2. Experimental procedure.- Experiments were carried out with wires (1mm in diameter, 40mm in length) of CENG pure iron, annealed 10 h at 400°C under purified hydrogen. Plastic deformations were performed in torsion at room temperature. Internal friction and microdeformation were measured with an inverted torsional pendulum, with a magnetic field of 200 Oe [8]. The internal friction spectra were made at about 0.8 Hz with an oscillating vibration amplitude $\epsilon_m = 10^{-5}$. We have verified that the static stresses used do not give permanent strains.

3. Experimental results

3.1 Internal friction Fig. 1 presents the internal friction spectra as a function of the plastic deformation amount. After mounting (curve 1), a broad peak is observed around 130K : this peak is labelled β_α according to the nomenclature of Ritchie et al. [6]. After 0.5 % deformation (curve 2), this peak grows strongly, shifts towards low temperature (115K) and presents a shoulder between 30 and 50K attributed to the α peak. For 2 and 4 % deformation, the β_α peak situated at 105K decreases and the peak α appears at about 30K and increases with the deformation amount. As observed by Dufresne et al. [7], the β_α peak temperature depends strongly on the vibration amplitude.

3.2 Microdeformation Figures 2, 3, 4 and 5 show the variation of micro deformation versus temperature according to the method used by Esnouf and Fantozzi [9] (the microdeformation plotted ϵ is corrected from the shear modulus variation with temperature $\epsilon = \epsilon_{\text{real}} \frac{\mu(T)}{\mu_0}$). When the static stress σ_s is applied at 4K, an elastic strain AB is observed. Then, during the linear heating, an extra amount of microstrain BC occurs. Subsequently, the specimen is cooled down to 4K (CD) and the static stress is removed : microdeformation recovered during the suppression of the stress is DE.

During a subsequent heating without stress, we observe a recovery of the strain (EF).

We have obtained similar results for different values of static stress.

The essential features of fig. 2 to 5 are as follows :

(1) - During the first heating with the static stress σ_s , microdeformation BC occurs in two stages : stage α between 10 and 50K and stage β between 70 and 140K. Stage α shifts towards low temperatures and increases strongly with the plastic deformation. The β component is lower than the α one and shows only a slight increase with the plastic deformation amount.

(2) - The recovery DE obtained by suppressing the stress at 4K is nearly equal to the elastic deformation AB.

(3) - During the successive heating without stress (EF), recovery of the strain occurs in two stages α_r and β_r . Stage α_r takes place at lower temperature than α stage and is more important for deformed specimens. Stage β_r occurs up to high temperatures and another recovery phenomenon takes place beyond about 200K. Recovery is complete towards 220K for non-deformed specimens and towards 250K for deformed specimens.

The derivative curves versus temperature are used to locate the α stage of microdeformation. Fig.6a shows the obtained results for the non-deformed specimen : we observe a big shift of the maximum (from 53K to 28K) when the bias stress increases. Fig. 6b shows the derived curves for a 2 % deformed specimen. Only a low

Fig. 1 : Internal friction spectra of Iron ($\epsilon_m = 10^{-5}$), (1) after mounting; (2) after 0.5 % plastic deformation at RT; (3) after 2% deformation; (4) after 4% deformation.

shift of the maximum is observed versus the bias stress. Furthermore, for all the deformed specimens, a substructure of the α stage occurs.

In order to determine the activation volume, we must normalize the microdeformation curves $\epsilon(T)$ [9]. From the observed shift of the curves versus static stress, we can obtain the activation volume from the following relation :

$$v = E / (\sigma - T \frac{d\sigma}{dT}) \quad \text{with } \epsilon = \text{constant}$$

The activation energy E for the α peak is about 0.06 eV [5]. The activation volumes measured for different amounts of plastic deformation and for a value of σ_s equal to about $4.5 \times 10^{-5} \mu$ are given by the following table.

plastic deformation	non-deformed	0.5%	2 %	4 %
v/b^3	60 ± 10	25	12	7 ± 5

We observe also a decrease of the activation volume when the static stress increases for the non-deformed specimen (from $60 b^3$ to $20 b^3$ when σ_s increases from $2 \times 10^{-5} \mu$ to $7.5 \times 10^{-5} \mu$).

4.- Discussion.-

α process : Microdeformation experiments are interesting because they can show an α component though no α peak is observed on the internal friction curve for the non deformed specimen. This result seems to indicate that in the very broad spectrum of β_α obtained for this specimen, the low temperature range of the spectrum is certainly linked to an intrinsic process α .

Stage α observed by microdeformation has the same characteristics than the one observed in f.c.c metals [9]: α stage takes place at higher temperature than the recovery stage α_r obtained without stress. The decrease of the activation volume when the plastic deformation increases and the importance of stage α_r should indicate that the local internal stress is large (much larger than in f.c.c. metals) and increases with the amount of plastic deformation. This conclusion is similar to the one of Astie' et al. [10] obtained from microdeformation experiments in the γ range

Fig. 2 : Microdeformation in iron without deformation with $\sigma_S = 3 \times 10^{-5} \mu_0$.

Fig. 3 : Microdeformation in Iron after 0.5 % deformation with $\sigma_S = 6 \times 10^{-5} \mu_0$

Fig. 4 : Microdeformation in iron after 2 % deformation with $\sigma_S = 6 \times 10^{-5} \mu_0$

Fig. 5 : Microdeformation in Iron after 4 % deformation with $\sigma_S = 6 \times 10^{-5} \mu_0$

The features of the α component are in good agreement with the double kink nucleation model on non-screw dislocations (71°). Therefore, these results confirm the given interpretation for the α peak in iron. Furthermore, the microdeformation curves always seem to present an α' component at low temperature, which must be precised.

β_α process : the β_α peak is generally attributed to the thermomechanical unpinning of non screw dislocations from impurity interstitials C and N. The microdeformation experiments show that β_α is not a simple breakaway process from pinning points situated on the dislocation line. Indeed, in this case, we should observe a microdeformation stage during heating with the static stress and a complete recovery of this deformation during the suppression of σ_s at 4K. In fact, no recovery is observed at 4K when the stress is removed (the AB deformation is equal to DE deformation). Therefore, dislocations remain pinned during the suppression of σ_s , pinning points being immobile at considered temperature. Thus, we can think that β_α is due to a dislocation depinning from an extended atmosphere. The microdeformation associated to the β_α peak occurs in a broad range of temperature as the β_α peak. It is difficult to observe a shift when the applied stress varies. This situation is not favorable to determine the characteristics of the β_α process but it is necessary to use a model of dislocation motion in dilute solid solution as proposed by Schlipf [11] and Ritchie [12].

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Fig. 6 : Derived microdeformation curves for iron.

Fig. 7 : Normalized microdeformation curves for iron.

- a) without deformation (1) $\sigma_s = 10^{-5} \mu_0$ (2) $\sigma_s = 3 \times 10^{-5} \mu_0$
 b) after 2 % deformation (1) $\sigma_s = 3 \times 10^{-5} \mu_0$ (2) $\sigma_s = 6 \times 10^{-5} \mu_0$