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ANALYSIS OF SNOEK-KOSTER (H) RELAXATION IN IRON

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Résumé - La relaxation Snoek-Köster due à l'hydrogène (S-K(H)) dans le fer est constitué de deux composantes à environ 110 K et 155 K (1 Hz). Nous avons étudié les effets non linéaires de cette relaxation S-K(H) et précisé ainsi les mécanismes responsables de chacun des processus.

Abstract - The Snoek-Köster relaxation due to hydrogen (S-K(H)) in iron shows two components at about 110 K and 155 K. The non-linear effects of this relaxation are studied and the mechanisms responsible for each component are specified.

I - INTRODUCTION

The S-K(H) relaxation is constituted of two components : SK-1 around 110 K and SK-2 at about 155 K (for a frequency of 1 Hz). We have previously studied the general features of these two components, particularly their behaviour during annealing /1,2/.

A complementarity between the S - K(H) relaxation and the α peak is observed /3,4/ and the (S - K)H relaxation and the γ peak can coexist in pure iron /2,5/. From these preceding results, the S-K(H) relaxation in iron has been attributed to the interaction of non-screw dislocations responsible for the α peak with hydrogen atoms.

The different models of the S-K relaxation did not allow to explain all the characteristics of this relaxation and we have proposed a modified model which attributes the S-K(H) relaxation to the thermally activated formation of double kinks in the presence of hydrogen atoms (SK-1) and of di-atomic hydrogen clusters (SK-2).

Nevertheless, to explicit the interpretation, we must specify the S-K relaxation behaviour, particularly the correlation between the heights of α and S-K(H) peaks, the broadening factors, the longitudinal or transversal drag of hydrogen atoms.

II - EXPERIMENTAL PROCEDURE

The experimental procedure is described elsewhere /2/. The specimens of CENG pure iron /6/ undergo the following treatments :

a) deformation by tension of 2 % and by torsion of 2 % at room temperature (T_a) ; hydrogen was charged by electrolytic method (5% H_2 SO_4 + CS_2) at T_a ; mounting in the pendulum

b) deformation by tension of 5 % and by torsion of 5 % at T_a and hydrogen charging at 250 K and mounting in the pendulum at this temperature.

III - RESULTS AND DISCUSSION

Firstly, we have studied the dependence of the height of the two components of the S-K relaxation with the vibrating stress amplitude, by decomposing the internal friction spectra. For the decomposition, we suppose the two components are asymmetric, the high temperature part being broader than the low temperature part. Thus, no fictitious peak appears. Furthermore, we assume that the broadening factor β and the asymmetry factor ϕ are constant. The values which give the best decomposition are indicated in table 1, as also the values of the activation parameters for the two components /4/.

Table 1

	E(eV)	τ_0 (s)	v/b^3	β	ϕ
SK - 1	0.22 ± 0.01	3.1×10^{-12}	10	2.5 ± 0.1	1.25 ± 0.05
SK - 2	0.37 ± 0.02	1.8×10^{-13}	10	3.1 ± 0.1	1.5 ± 0.05

Fig. 1 shows the variation of the peak height as a function of the vibrating strain amplitude. The SK-1 component increases slightly up to 2×10^{-5} then decreases slowly. This behaviour is not linked to the Paré condition, the internal stresses being important but is due to the dragging of the hydrogen atoms. Indeed, when the stress increases, the dislocation speed must increase to sweep a higher area. For a given temperature, the dislocation speed is limited by the hydrogen diffusion and cannot increase ; so the area swept by the dislocation increases more slowly than the strain amplitude and the internal friction decreases.

The SK-2 component is very sensitive to the strain amplitude because the active dislocation length is higher than for the SK-1 component /2/. Furthermore, the possibility of a breakaway process cannot be excluded.

Secondly, we have studied the effect of a bias stress on the internal friction spectrum. The procedure is the following : i) the IF spectrum is measured with a vibration strain amplitude $\epsilon_m = 5 \times 10^{-6}$ (fig. 2a) ; ii) at 4 K, a bias stress $\sigma_S = 10^{-4} \mu$ is applied and the IF is measured with the same strain amplitude ϵ_m (fig. 2b) ; iii) from 240 K to 4 K, the bias stress is maintained and the IF is measured during heating with σ_S (fig. 2c) ; iii) σ_S is removed at 240 K and the IF spectrum is determined.

For the two treatments, we observe that the SK-1 component increases slightly and shifts weakly towards low temperature and there is no difference between the curves b and c of fig. 2. This behaviour is probably due to the participation of shorter segments of dislocations when the bias stress is applied.

The SK-2 component is strongly developed by the bias stress during the first application (fig. 2b) then decreases during the second run with the stress. Furthermore, the peak temperature is increased perceptibly.

When the bias stress is applied at 4 K, the hydrogen clusters are not mobile and the dislocation position does not change. During the first heating, the clusters become mobile in the SK-2 temperature range and the dislocation reaches a new equilibrium position, sweeping an important area ; so, the IF peak is higher (fig. 2b). When the dislocation reaches its new equilibrium position, the height of the barrier for the double kink formation increases and the peak height decreases and shifts towards high temperature. The hypothesis of Ritchie /7/ for the S-K relaxation (lateral dif

fusion of hydrogen along the dislocation) cannot be retained, the two components being always present with the bias stress (fig. 2b and 2c). Finally, the problem of the complementarity of the S-K relaxation and the α peak must be resolved. Schoeck /8/ notes that for the kink model the S-K(H) relaxation strength must equal the initial α peak height. This assertion is not correct. Indeed, the microcreep experiments show that the α microdeformation stage is equivalent to the S-K(H) one. For the IF measurements, we must take into account the broadening factor of the peak : we can consider that the product of the peak height δ by the broadening factor β is constant for the two peaks, the two processes being similar /9/.

Our results agree with this hypothesis in the two cases (two components SK-1 and SK-2 or only the SK-2 component) as shown by table 2 and confirm the kink model and the complementarity between the α and S-K(H) relaxation.

Table 2

	α	SK-1 + SK-2	SK-2
δ (micro)	5.1×10^{-2}	$4.5 \cdot 10^{-2}$	$4.7 \cdot 10^{-2}$
δ (I.F.)	4.8×10^{-3}	$6.5 \cdot 10^{-3}$	$12 \cdot 10^{-3}$
δ (I.F.)	8.5	6.1	3.7
δ (I.F.) $\times \beta 10^3$	40.8	39.6	44.4

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Fig.1.- Evolution of the relaxation strength for the SK-1 ($\bullet$) and SK-2 ($\blacklozenge$) components of the S-K relaxation as a function of the vibrating stress amplitude.

Fig.2.- I.F. spectra for an hydrogen charged specimen with a vibrating strain amplitude = $5 \times 10^{-6} \mu$:
a) Without bias stress; b) with a bias stress $\sigma_s = 10^{-4} \mu$ applied at 4K ; c) with a bias stress applied at 240K