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THE SNOEK-KÖSTER RELAXATION IN IRON

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Abstract.—An investigation on the Snoek-Köster peak in high purity iron doped with 1000 at ppm of carbon was carried out. The study of the influence of low temperature deformation on the S-K peak revealed that screw dislocations may not play the major part in the S-K relaxation in α-Fe. The carbon S-K peak's parameters were found to be $H_{SK} = 1.84 \pm 0.03$ eV and $\tau_{SK} = 0.7 \times 10^{-19}$ s$^{-1}$. It was also found that carbon-vacancy pairs might influence the S-K relaxation. However these experiments are not considered as an evidence in support of Mondino and Seeger's interpretation. The $\beta_2$ and $\beta_\gamma$ peaks were also observed.

Introduction.—In order to explain the Snoek-Köster (S-K) relaxation in b.c.c. metals Schoeck model, for a long time, has been widely accepted. Actually, this relaxation process was supposed to be caused by the motion of non specified dislocations controlled by the diffusion of interstitial atoms (C, N or O) [1]. However a few difficulties arose in accounting for some striking experimental results concerned with carbon S-K peak in α-Fe. It has been claimed [6] that interstitially dissolved carbon atoms do not contribute to the S-K peak. In addition, they retard its formation or even completely suppress this effect because of precipitation on the dislocations. Puzzling results have been obtained, so far, on the question of carbon S-K peak in iron. Some results confirmed the existence of this peak [4,5,7] while the others do not [6,8,9]. In fact, this problem has been explained by Mondino and Seeger [10] in terms of the interaction of carbon atoms with vacancies. Since, not only are dislocations generated by cold work but vacancies as well the formation of C-V pairs and it's high dissociation activation enthalpies $H_{diss}^{CV} = 1.6$ eV [11] may cause that the carbon S-K peak is not fully developed or even might be undetectably small during the first warm-up. Consequently, as suggested by Mondino, full development of the carbon S-K relaxation requires annealing above the peak temperature i.e. after dissociation of C-V pairs. Nevertheless, there is no experimental evidence to bear out this hypothesis [9].

According to a recent theory of Seeger [12], the S-K relaxation in b.c.c. metals is attributed to the formation of kink pairs in screw dislocations in the presence of high local concentration of foreign interstitial atoms. This signifies that the activation enthalpy of the S-K relaxation is to be the sum of the kink pair formation enthalpy in $a_0/2$ $<111>$ screw dislocations $2H_K$ and the migration energy...
of the foreign interstitials. Actually, if the $a_g/2 <111>$ screw dislocations are really involved in the S-K relaxation one would expect higher relaxation strength when plastic deformation is performed at low temperatures, compared with the same amount of room temperature (RT) deformation. It is to be noted that both theories explain the experimental results fairly well.

Taking into account the problem of transient effect in the S-K relaxation of C in α-Fe and the last theory of the S-K peak this prompted us to start the experiments described in this paper. Simultaneously, the $\beta_2$ and $\beta_\gamma$ peaks have been observed. In the following, we first discuss the problem of transient effect of C and the influence of low and room temperature deformation on the S-K peak in α-Fe doped with 1 000 at ppm of C. In the last part we shall briefly deal with the $\beta_2$ and $\beta_\gamma$ peaks.

Experimental procedure. - The samples are 0.6 mm diameter wires, 100 mm in length, of high purity CEN-G iron [3] doped with 1 000 at ppm of carbon. The heat treatment included 5 hr anneal at 823 K in an atmosphere of helium followed by quench in a flow of helium gas. The quenching cooling rate thus was approximately 500 K s$^{-1}$. After this solution heat treatment the samples were quickly transferred into liquid nitrogen where they were stored until deformation procedure. Mixed deformations in tension were employed, namely: 5 % at RT + 5 % at 77 K, 7 % at RT + 3 % at 77 K, 9 % at RT + 1 % at 77 K, 10 % at RT and also directly 2 % at 77 K and 2 % at RT. Then additional deformations in torsion of 3.5 % at 77 K and 6 % at 77 K were performed in the pendulum after getting the S-K peak caused by the previous deformation in tension. Internal friction and square of vibration frequency were simultaneously measured using an automatic inverted torsion pendulum operated near 1 Hz and over a temperature range from 100 to 673 K during linear warm up with a heating rate of 200 K h$^{-1}$. The specimen was subjected to a mean surface strain amplitude of $9 \times 10^{-6}$ and immersed in a magnetic field of 100 Oe to suppress magnetoelastic phenomenon.

The precise values of the S-K peak's parameters and its shape have been determined after substraction of the background according to the relationship

$$Q_{\text{Backg}}^{-1} = A + B \exp(-\frac{H}{kT}),$$

where $A$, $B$ and $H$ and the parameters to be determined after optimisation of the $\chi^2$ function. An appropriate number of experimental points, on both sides of the peak, are taken into computation. The $\beta$ parameter of lognormal distribution has been estimated according to the formulas given by Nowick and Berry [2].

Experimental results. - Fig. 1 shows the dependence of the S-K peak after background substraction; in each case the total plastic deformation of the specimen attained 10 % but with different ratio of CW at RT and CW at 77 K (curve a). After additional CW at 77 K of 3.5 % and 6 % in torsion the dependence of the S-K peak is presented on curves (b) and (c), respectively. The following observations may be made:
(i) With increasing degree of CW at 77 K the S-K peak is decreasing.
(ii) The difference between $Q_{\text{max}}^{-1}$ after 10 % at RT and 9 % at RT + 1 % at 77 K is always negligible.
(iii) The aforementioned relationship is valid for additional CCJ at 77 K of 3.5 % and 6 %.

This experimental part of the work was repeated twice so as to be sure of getting a correct answer on the question of the influence of low temperature cold work on S-K peak. The results were exactly the same.

We must notice, too, that after a small deformation of 2 % at 77 K and 2 % at RT the S-K peak is very slightly different, indeed. The exact values are the following: for deformation at RT $Q_{\text{max}}^{-1} = 4.4 \times 10^3$, $T_{\text{max}} = 558$ K, $\beta = 3.5$ and for deformation at 77 K $Q_{\text{max}}^{-1} = 4.0 \times 10^3$, $T_{\text{max}} = 556$ K, $\beta = 3.5$. Generally, on the first run after deformation in tension a Snoek peak with a very small contribution on both sides is observed. Then, starting from 450 K a peak is rising; it's maximum varies within a temperature range of 530-600 K depending on the deformation (Fig. 2a, b, curves 1). Immediately after reaching 673 K a measurement is continued with cooling down (curves 2). The cooling rate is also 200 K s$^{-1}$. In this case, an extremely stable peak at 545 K ($f = 1$ Hz) is observed. This is a carbon Snoek-Köster peak in α-Fe. Several runs up and down may be performed and the S-K peak is unbelievably repeatable within an error in an estimation of $Q_{\text{max}}^{-1}$ and $T_{\text{max}}$ of 1 %.

The changes in internal friction and modulus are very similar after deformation performed in all "mixed" deformation and after additional cold work at 77 K with the only one exception of the so-called S-K peak on the first run when warming-up after deformation in tension (Fig. 2a, b, curves 1). For this reason only one more typical curve is to be presented. Fig. 3 illustrates characteristic spectra of a specimen deformed 10 % at RT plus additional 3.5 % in torsion at 77 K. The 270 K and 350 K peaks are usually observed. The S-K peaks in the up (curve 1) and down (curve 2) runs are very similar. In the later case, however, high temperature background is lower and perfectly flat spectrum below 400 K is observed. High temperature background damping removal and a real S-K peak is also presented. Moreover, a pinning stage caused by carbon atoms starts at about 320 K after all variants of "mixed" cold work in tension. After additional CW at 77 K this stage starts below 300 K. This is to emphasize the coexistence of all the already mentioned peaks, namely: 270 K, 350 K, the S-K peak and the pinning stage in the up runs after additional CW in torsion at 77 K.

The 270 K and 350 K peaks occur only after additional deformations at 77 K when fresh segments of screw dislocations are created. Although a detectable increase of 270 K peak is observed after deformation 5 % at RT + 5 % at 77 K. The 270 K peak starts rising from 110-120 K, it's maximum is always at 270 K and does not effect any change on the modulus curve. During annealing at it's peak temperature for 5 min, the peak is decreased by 50 % and a small increase in modulus is observed. In the case of 350 K peak an annealed at 350 K for 5 min. cause the same effect, but increase in modulus is 3 times greater. Fig. 3 (curve 1) shows the effect of
annealing at 370 K for 8 min.

Discussion.— In the present investigation after different kinds of 10 % cold work and 2 % cold work of pure iron doped with 1 000 atm ppm of carbon the Snoek-Köster peak is not fully developed in the first run up, but when cooling down. This peak is highly stable and it's shape and β parameter is exactly the same when second and following runs are made, and also after ageing at 673 K for 1 hour in absence of Snoek peak. The absence of the entire S-K peak in the first run ups and it's appearance when cooling down i.e. when 673 K was reached can be explained in terms of carbon-vacancy interaction (for details see [19,20]). In fact, fully developed S-K peak may also be obtained when the highest temperature during run ups was 600 K; this temperature is sufficient for dissociation of C-V pairs. Finally, released carbon atoms do take part in the S-K relaxation. These results are in agreement with the interpretation of Mondino and Seeger [10].

It is also confirmed that the peak temperature is decreasing when the degree of CW is increased [6]. After deformation of 2 % at RT the \( T_{\text{max}} = 557 \text{ K} \) while after 10 % at RT \( T_{\text{max}} = 545 \text{ K} \).

Thanks to the stability of the S-K peak we determined the activation energy, however, in a comparatively small frequency range of 0.3-1.5 Hz. Thus, the activation energy is \( 1.84 \pm 0.03 \text{ eV} \) and the pre-exponential factor is \( 0.7 \times 10^{-19} \text{ s}^{-1} \).

Let us now consider the Seeger's hypothesis, according to which, in \( \alpha\text{-Fe} \) the S-K relaxation is caused by the kink-pair formation in screw dislocations in the presence of a high local concentration of carbon atoms (here in this study). The data reported (Fig. 1) suggest that it is not this case. Moreover, when the amount of 77 K deformation is increased the S-K peak is decreased. Before definite conclusions can be drawn, however one must take into account comparatively small changes in the \( Q^{-1}_{\text{max}} \) of the S-K peak after deformation of 10 % at RT and 5 % at RT + 5 % at 77 K. Virtually, it is 18 % decrease. The same ratio is valid for additional cold work in torsion at 77 K.

Albeit, we have to resort to another experimental data on Fe-40 ppm N by Ino and Sugeno [13]. They showed that the S-K peak is drastically decreased when deformation temperature is decreasing. For the sake of clarity, let us give the values of the S-K peak after deformations at 473, 303, 203 K i.e. \( Q^{-1} = 8.9 \times 10^{-3} \), \( Q^{-1} = 4 \times 10^{-3} \), \( Q^{-1} = 1.2 \times 10^{-3} \), respectively. In view of the above it seems unlikely that \( a_{0}/2 <111> \) screw dislocations play the major part in the S-K relaxation in \( \alpha\text{-Fe} \) in both systems Fe-C and also in Fe-N.

The 270 K and 350 K peaks in high purity iron doped with carbon may be identified as \( \beta_{2} \) and \( \beta_{Y} \) peaks as in pure iron [14,16]. In accordance with these results the \( \beta_{2} \) peak has to involve screw segments of dislocations and intrinsic point defects created by cold work performed directly at 77 K or with prestrain at RT (Fig. 2b, 3). To observe \( \beta_{Y} \) peak the presence of fresh dislocation is clearly needed. In order to generate both of these peaks a minimum 1 % deformation in torsion at 77 K has to be
employed. Detailed consideration on the mechanism of the $\beta_2$ and $\beta_Y$ peaks will be described elsewhere [17].

**Conclusions.** Within the scope of this present investigation it is concluded that:

1. It is confirmed that the existence of C-V pairs mask the Snoek-Köster relaxation in $\alpha$-Fe. Although, after dissociation of C-V pairs released carbon atoms do take part in the S-K relaxation and a symmetrical S-K peak is observed.

2. The experimental results on S-K relaxation in Fe-C and Fe-N systems are not in agreement with the Seeger's theory on the S-K relaxation in b.c.c. metals. It is to be pointed out that when the deformation temperature is decreased or the amount of cold work at 77 K is increased the height of the S-K peak is decreasing. However, further supporting evidences are desired since these results are not consistent with recent results on Nb-O system [18].

3. It is also worthwhile to point out that the S-K peak may be observed when there is no carbon atoms in solid solution since no Snoek peak was detected after the first run. It is therefore expected to consider all carbon atoms in an extended Cottrell atmosphere.

4. A detailed analysis should be carried out for the 523 K peak in ferrous martensite. It is probably safe to assume that the S-K peak and 523 K peak in ferrous martensite might be caused by a nearly similar relaxation process.

To clarify the interpretation of the S-K mechanism further work is clearly necessary.

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**References**


Fig. 1 - The dependence of the S-K peak's height as a function of cold work performed at RT and 77 K. Total deformation was 10%, curve a. Curves b and c correspond to additional deformation of 3.5% and 6% in torsion at 77 K, respectively.

Fig. 2 - Internal friction spectrum and square of the frequency as a function of temperature for a specimen deformed 10% at RT (figure a) and 5% at RT + 5% at 77 K (figure b). Curves 1 correspond to the first run-up while curves 2 to the first run-down.

Fig. 3 - Internal friction spectrum for a specimen predeformed 10% at RT with additional deformation of 3.5% at 77 K in torsion. Curve 1 - run-up, curve 2 run-down; --- computed background, ---- real S-K peak.