THE SNOEK-KÖSTER RELAXATION IN BODY-CENTRED CUBIC METALS

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Résumé - Dans les expériences de frottement intérieur sur le fer α et les métaux du groupe transitoire V contenant des atomes lourds (O, N, C) en solution solide interstitielle (ISA) des pics de Snoek-Köster (SK) apparaissent, après écrouissage, à des températures beaucoup plus élevées que les pics de Snoek des ISA. La position et la hauteur du pic dépendent du degré d'écrouissage et du teneur en ISA. Des expériences sur des monocristaux de Nb-O, Ta-O et Fe-N montrent clairement que deux processus de relaxation SK-1 (pic d'écrouissage "classique") et SK-2, existent. On peut comprendre la plupart des résultats expérimentaux avec un modèle de dislocation fondé sur un "dragging" des ISA mobiles par des boucles de dislocations agitées par la contrainte. Dans une théorie plus raffinée, la relaxation de Snoek-Köster dans les métaux c.c. est attribuée à la formation thermiquement activée des paires de décrochements sur les dislocations vis $a/2 \langle 111 \rangle$ en présence des ISA. Les conceptions élémentaires et les paramètres atomiques de cette théorie sont en bon accord avec les faits expérimentaux; particulièrement elle est en mesure d'expliquer pourquoi le pic SK-2, à température plus élevée, peut posséder une énergie d'activation plus faible que le pic SK-1, qui apparaît à plus basse température.

Abstract - In internal friction experiments on α -Fe and group-V transition metals containing heavy interstitial solute atoms (ISA) (O, N, C) Snoek-Köster (SK) peaks appear after cold work at temperatures substantially higher than the ISA Snoek peaks. Peak position and height depend on degree of cold work and ISA content. Experiments on monocrystals of Nb-O, Ta-O and Fe-N clearly indicate that there exist two relaxation processes SK-1 (the "classical" cold-work peak) and SK-2. Most experimental results may be understood with a dislocation model based on dragging of mobile ISA by dislocation segments moving under stress. In a more refined theory the SK relaxation in bcc metals is attributed to the thermally activated formation of kink pairs in $a/2 \langle 111 \rangle$ screw dislocations in presence of ISA. Basic assumptions and atomistic parameters within this kink theory are in good agreement with experimental facts; it is especially able to explain that the high temperature SK-2 peak may have a lower activation enthalpy than the low temperature SK-1 peak.

1. Introduction

Dislocations in metals give rise to different types of anelastic relaxation. The intrinsic properties of dislocations which correlate with

the crystal structure, and which lead e.g. to the well known Bordoni relaxation, were treated in recent reviews /1,2,3/. Additional relaxation phenomena occur due to the interaction of dislocations with defects of atomic size, i.e. point defects. These interactions may be classified into two categories:

- (i) Interactions with intrinsic point defects produced e.g. by cold work (at low temperatures) or irradiation. To these belong the relaxations of the Hasiguti type (see e.g. /4,5,6/).
- (ii) Interactions with foreign atoms. In bcc metals heavy interstitial solute atoms (ISA) such as C,N,O exert a strong influence on dislocations due to their local, anisotropic strain field. This leads to relaxation phenomena which have long been known as "cold-work" peaks. Recently the nomenclature Snoek-Köster (SK) relaxation /4/ has become more usual (to differentiate it e.g. from other "cold-work" effects). A common feature of all SK relaxations is that mobile ISA are involved. They require a dislocation "dragging" mechanism as was first recognized by Schöck /7/ (see below). The SK relaxations of heavy ISA in bcc metals occur at substantially higher temperatures than the corresponding ISA Snoek peak temperatures (see e.g. /3,4,8/). By this fact the SK relaxations may be distinguished from intrinsic dislocation relaxations which occur at substantially lower temperatures.

In this survey hydrogen SK relaxations are excluded. For group V metals (high solubility for H and other ISA) the α -peaks were considered erroneously for some time as being an intrinsic dislocation relaxation. Recently it was recognized that they are of SK-H type and masked the real intrinsic dislocation relaxations (see e.g. reviews /1,2,3/).

The Snoek-Köster relaxation was first reported by Snoek /9/ in internal friction (IF) experiments on iron which was loaded with nitrogen after cold rolling. Fig. 1 shows Snoek's first measurement. An IF peak appeared at ≈ 450 K ($f = 0.2$ Hz), i.e. substantially higher than the corresponding N Snoek peak (≈ 285 K). Later experiments of Kê /10/ showed the same phenomenon. Köster, Bangert and Hahn /11/ were the first to study systematically the influence of cold work, carbon and nitrogen contents, and annealing treatments. Fig. 2 has been taken from their work and shows the influence of different degrees of deformation. Köster et al. concluded that the phenomenon is due to an interaction of carbon and nitrogen (i.e. ISA) with dislocations. For these historical reasons the nomenclature "Snoek-Köster" relaxation was suggested /4/. The SK relaxation should not be restricted to iron but should also occur in other bcc metals, especially group V metals which exhibit a relatively high solubility for O and N. This was first shown by experiments on Ta and Nb /12-14/.

In the following experimental part (2) we try to summarize the experimental situation for iron and group V metals. Theoretical aspects are treated in (3). Earlier surveys were given in books /4,5,8/.

2. Experimental Situation

2.1 α -Iron

First IF experiments on the SK relaxation in α -Fe /9,10/ were carried out after doping with nitrogen, i.e. they showed the nitrogen SK relaxation. In the investigations of West /15/ and Köster et al. /11/ low alloy steels containing C and N were used. By this it is not possible to distinguish from these experiments if N and C (or only N) caused the relaxation phenomena. Kamber, Keefer and Wert /16/ first showed

Fig. 1: Snoek's first IF curve on deformed Fe with 0.25 at.% N after cold rolling /9/ ($f = 0.19$ Hz).

Fig. 2: Influence of different amounts of plastic deformation on the IF of iron ($f \approx 1$ Hz) /11/.

Fig. 3: Survey of the relaxation processes in deformed Fe-N and Fe-C between 415 K and 1000 K (Arrhenius diagram of relaxation times).

that for iron doped with C the corresponding carbon SK relaxation exists at about the same temperature as for N. The peak height for the carbon peak was, however, less pronounced and developed only after heating to temperatures above the peak position in temperature. This was later confirmed by Barrand and Leak /17/, Petarra and Beshers /18/ and Ferron et al. /19/.

The Arrhenius diagram of Fig. 3 ($\ln \tau$ vs. $1/T$) gives a survey on the experiments carried out on plastically deformed iron for the temperature range from 415 K to 1000 K. ^{+) Measurements on Fe doped with N are represented by open symbols, on Fe-C by filled symbols. For samples}

Fig. 4: IF of Fe with 0.1 at.% C after 10% deformation at RT and 3.5% at 77K. 1: run up, 2: run down ($f \approx 2$ Hz) /21/.

Fig. 5: IF of Fe with 0.045% N (curves 1 and 5) and with 0.06% N (curves 2-4) before (1) and after different degrees of plastic deformation (2a,b: 5%, 3a,b 7.5%, 4a,b 10%; 5: 25% - at RT) ($f \approx 2$ Hz) /23/.

which contained C and N we used hatched symbols. Experiments which comprehended different degrees of deformation are represented by horizontal dashed lines with bars indicating the temperature range of the peaks. Measurements on single crystals are characterized by encircled symbols. +)

Published values for the relaxation parameters, H and τ_0 , (according to $\tau = \tau_0 \exp(H/kT)$) are represented in Fig. 3 by inclined, full lines (the frequency range covered is indicated by bars, if not known by arrows). We restrict the discussion to experiments which used samples which contained only C or N (doped samples). For Fe-N Petarra and Beshers /18/ published $H = 1.65$ eV, $\tau_0 = 10^{-18}$ s and Buchmann and Kennedy /20/ 1.91 eV, $\tau_0 = 3.3 \cdot 10^{-21}$ s. For Fe-C there was published to our knowledge only one value by Magalas et al. /21/: $H = 1.84$ eV, $\tau_0 = 7 \cdot 10^{-19}$ s (the authors specified $\tau_0 = 0.7 \cdot 10^{-19}$ s; this is probably a typing error since this Arrhenius line does not fit their experimental point). There exist only a few experiments on monocrystals from Ino and Sugeno /22/ and recently from Tietze et al. /23/, both Fe-N.

As can be seen from Fig. 3 the low frequency IF experiments - only the experiments of West /15/ represent mechanical after-effect measurements - on deformed Fe-C and Fe-N show deformation induced peaks (known as SK peaks) in the temperature from about 450 K to 620 K. Apparently the SK-peaks for C and N are situated in about the same temperature range. Differences between measurements of different authors are probably due to (i) different C and N contents (total and in interstitial solution), (ii) different degrees of plastic deformation and (iii) different (overall) purity of the iron samples. Most (probably all) iron samples used up to the end of the seventies contained a rather high amount of metallic impurities (up to several hundred at.ppm), samples from low alloy steels even more.

A new generation of experiments which used high purity iron started only recently. As we shall see below a similar situation exists for

+) τ -values calculated from the peaks in IF curves published in literature according to $\tau = 1/2\pi f$ (f = measuring frequency). The dotted horizontal line in Fig. 3 is for $f = 1$ Hz ($\tau = 0.16$ s).

group-V transition metals. Magalas et al. /21/ studied the SK relaxation in high purity Fe doped with 0.1 at.% carbon. Fig. 4 shows measurements on a sample deformed 10% at room temperature and additionally 3.5% at 77 K. A stable, well pronounced peak appears at 545 K. Recently Weller, Tietze, Diehl and Seeger /23/ published measurements on monocrystals of high purity Fe which were doped with nitrogen. Fig. 5 shows measurements on two monocrystals which contained 0.045 and 0.06 at% N and which were subjected to different amounts of plastic deformation. As can be seen from Fig. 5 two deformation induced peaks appear in the temperature range from 550 K to 800 K, designated as peak (1) and (2) in Fig. 5. With increasing degree of deformation both peaks increase and are shifted to lower temperatures. Peak (1) is less stable against annealing than peak (2) as is shown in Fig. 5 by subsequent runs for the same degree of deformation.

The data from /23/ are included in the Arrhenius diagram in Fig. 3. Deformation induced peaks (i.e. peaks which increase with degree of deformation) above 700 K were already reported earlier for polycrystalline iron /24,25,26/. However, they were interpreted as being due to a grainboundary relaxation mechanism as suggested by Kê /10/. As far as we know, peak (2) in /23/ is reported for the first time for deformed Fe-monocrystals. In this light its earlier interpretation as a grain-boundary sliding mechanism must be doubted.

In Fig. 3 not all measurements obtained on deformed ferritic steels could be included. Additional studies exist on plain carbon or alloyed steels with C, in which martensitic structures exist after quenching. These steels show IF peaks between about 470 K and 570 K - even without cold work (see e.g. /27,28,29/).⁺

2.2 Group-V transition metals

The experimental situation for Nb and Ta was recently discussed in detail /30/. So we confine to summarize the essential points.

2.2.1 Niobium

The Arrhenius diagram of Fig. 6 shows published measurements on deformed niobium together with recent experiments obtained for Nb-O /30/. Additionally the Snoek relaxations for O, C and N in Nb (acc. to /30/) are represented (long, thin lines)⁺⁺. Measurements from /30/ obtained for a deformed Nb-O (100 at.ppm) monocrystal are shown as thick lines, experiments on two O-doped polycrystals (Nb-A, Nb-B) with intermediate lines (for details see /30/). Stress relaxation experiments by Oytana and Varchon /31/ are presented by dashed lines (the thin line corresponds to an extrapolation to zero annealing time, the thicker one to annealing to 418 K for 70 h). The (dotted) horizontal line represents

⁺) Apparently dislocations originating from martensitic structures give rise to relaxations which may be of SK-type. Due to the relatively complicated structure of these samples it may be understood that abnormally low values for the activation enthalpy are sometimes reported (e.g. $H = 0.5$ eV, $\tau_0 = 10^{-6}$ s /28/).

⁺⁺) For Nb and Ta it has turned out as useful to include the Snoek relaxation data /30/. In literature sometimes SK relaxations were confused with Snoek relaxations (especially when the shift of the peak temperature with measuring frequency were not considered). Earlier data on the carbon Snoek relaxation in Nb have to be revised /30, 46/.

Fig. 6: Arrhenius diagram ($\ln \tau$ vs. $1/T$) for relaxation processes in Nb between 300 K and 1000 K.

Fig. 7: IF of a Nb monocrystal with 100 at.ppm O as a function of successive plastic deformation /30/. The Q^{-1} scale above 600 K has been increased by a factor of 2.5.

- 1 — before plastic deformation ($f = 1.20$ Hz)
- 2 — after 0.6% torsion ($f = 1.21$ Hz)
- 3 — after additional 1.5% tension ($f = 1.21$ Hz)
- 4 — after additional 0.3% torsion ($f = 2.8$ Hz).
- 5 after total plastic deformation of 0.6% in torsion + 1.5 in tension + (0.3% + 0.6%) in torsion ($f = 4.9$ Hz).

a measuring frequency of 1 Hz. In Fig. 6 all individual measurements which are represented by filled symbols by Boone and Wert /13/ van Ooijen and van der Goot /32,33/ and Schlät /34/ are attributed to the SK-relaxation of nitrogen. An Arrhenius fit yields $H = 1.7$ eV, $\tau_0 = 3.2 \cdot 10^{-12}$ s (dotted line).

All data points from literature which we correlate with the oxygen SK relaxation in Nb are represented as open symbols. These may be subdivided into two groups. One group of measurements for $f \approx 1$ Hz is situated at lower temperatures at about 480 K - 600 K: de Lamotte and Wert /14/ Gebhardt and Rothenbacher /35/ and Igata et al. /36/. On the other hand van Ooijen et al. /32,33/ claimed that the O-SK relaxation should occur at higher temperatures (≈ 700 K). Schlät /34/ observed two peaks at 600 K and 800 K ($f = 8$ Hz). Zolotukhin et al. /37/ investigated the IF of niobium (and Ta) vacuum condensates (which usually exhibit a relatively high dislocation density) in the kHz-range. A peak at ≈ 760 K which appears in the first IF heating curve correlates with the low temperature group of measurements for the oxygen SK relaxation in Fig. 6 for $f \approx 1$ Hz.

The controversy in literature whether the oxygen SK relaxation in Nb may be located at lower (≈ 500 K) or higher (≈ 700 K) temperature was recently clarified by experiments on ultrapure Nb which was carefully doped with only oxygen /30/ (in Fig. 6 thin lines for samples Nb-A and Nb-B, thick lines for a Nb monocrystal; for details see /30/). These experiments showed that there exist two deformation induced peaks in Nb (designated (1) and (2) in Fig. 6). Fig. 7 shows these IF measurements on a Nb-O monocrystal from ultrapure Nb doped with 100 at.ppm O, which was subjected to different degrees of plastic deformation /30/. Two peaks designated as (1) (at ≈ 545 K for $f = 1.2$ Hz) and (2) (at ≈ 730 K for $f = 1.2$ Hz) can be seen. The heights of both peaks increase with degree of deformation. (The shift in peak temperature is caused by different measuring frequencies between 1.2 and 4.9 Hz). For peak (1) and (2) the following relaxation parameters were determined: (1): $H = 2.00$ eV, $\tau_0 = 4.2 \cdot 10^{-20}$ s; (2): $H = 1.68$ eV, $\tau_0 = 3.4 \cdot 10^{-13}$ s. It is remarkable that the activation enthalpy of the low temperature peak was found to be higher than that of the high temperature peak.

2.2.2 Tantalum

Fig. 8 shows the Arrhenius diagram for measurements obtained on deformed tantalum above 400 K. Again the Snoek relaxations for O, N and C in Ta are included (long, thin lines, acc. to /30/). All data for temperatures above about 550 K which are represented by open symbols we assign to the oxygen SK relaxation. To these belong the data from Schöck and Mondino /12/ and de Lamotte and Wert /14/ as well as one point from van Ooijen et al. /32,33/ which was assigned in our opinion erroneously to the nitrogen Snoek relaxation (this point lies sufficiently far away from the N-Snoek line). Probably the IF peak observed by Delobelle et al. /39/ which was attributed to the carbon Snoek relaxation is in reality an oxygen SK relaxation. The data marked in Fig. 8 by full symbols /32,33/ belong in our opinion to the nitrogen SK-relaxation.

Experiments on ultrapure tantalum which was only doped with oxygen were recently performed in Stuttgart /30,40/. Measurements on polycrystalline Ta doped with about 700 at.ppm O and after 30% deformation (sample Ta-A) and after 5% deformation (Ta-B) are shown in Fig. 8 by thick full lines /30/. These experiments show that the assignment of the open symbol points to the oxygen SK-relaxation is justified. Monocrystals of high purity Ta which were doped with different amounts of oxygen were studied by Rodrian and Schultz /40/. Their data on "pure"

Fig. 8: Arrhenius diagram ($\ln \tau$ vs. $1/T$) for relaxation processes in Ta between 400 K and 1000 K.

Fig. 9a: IF ($f \approx 1.3$ Hz) of Ta with 25 at.ppm O after plastic deformation and subsequent annealing treatments (for details see /40/).

Fig. 9b: IF ($f \approx 0.8$ Hz) for Ta with 167 at.ppm O after plastic deformation (curve 1) and after annealing to 730 K (curve 2) /40/. The O-Snoek peak of undeformed Ta is shown in addition.

Ta (≈ 5 at.ppm O) may be correlated with stress relaxation experiments on Ta-O from Oytana /41/. Fig. 9 shows IF curves from Rodrian and Schultz /40/ for a Ta-monocrystal doped with 25 at.ppm O (Fig. 9a) and a monocrystal containing 167 at.ppm O (Fig. 9b) after plastic deformation. The oxygen SK peaks are located at 600 K (9a) or 660 K (9b). (The Snoek peak which is increased by deformation is designed by these authors as " γ_2 "). The SK peak in Fig. 9b for Ta-O (167 at.ppm) which correlates well with polycrystalline samples Ta-A and Ta-B (see Fig.8) is superimposed to a rapidly increasing background. In our opinion this belongs to a second deformation induced peak in Ta-O which is situated at higher temperatures. This peak has not been measured up to now on monocrystals.

That is we expect that the same situation exists for Ta-O as for Nb-O, i.e. two deformation induced SK-peaks. For the low temperature peak (1), studied up to now, good coincidence exists between experiments on ultrapure poly- and monocrystalline Ta-O /30,40/ and earlier experiments as can be seen from Fig. 8.

2.2.3 Vanadium

To our knowledge only unpublished IF measurements of Mondino et al. /42/ exist on cold-worked vanadium. Their samples contained oxygen in excess to nitrogen. The IF curves at the first heating up after cold-work showed a peak at about 660 K ($f \approx 1$ Hz) which was decreased drastically by subsequent annealing to about 720 K.

Presumably this IF peak at 660 K can be assigned as oxygen SK-1(?) relaxation in deformed vanadium.

Experiments on V suffer from the difficulties (i) in producing V-samples with low content of metallic impurities, and (ii) in doping with only one species of ISA, O or N. The latter one is necessary to distinguish clearly between O and N SK relaxations since the Snoek peak temperatures for O and N in V ($f = 1$ Hz) are only about 85 K apart from each other /5/ (compared with 140 K for Nb and 196 K for Ta, see e.g. /30/), i.e. the migration enthalpies of O and N in V are close together.

2.3 Group-VI transition metals

As far as we know there exist no experiments on SK relaxations in deformed Cr, Mo or W (group-VI transition metals). The reason may be that these metals exhibit, in contrast to group-V metals an extremely low solubility for ISA (O,N,C). Even the location of the Snoek peaks in these metals is not without doubt as recent controversies on Mo /43,44/ show. An additional handicap for the investigation of possible SK relaxations in these metals is their tendency to brittleness after doping with ISA. For chromium, which becomes extremely brittle by small amounts of oxygen, even the intrinsic dislocation relaxations are unknown; in addition there is strong interference in IF experiments with effects resulting from its antiferromagnetic nature (see e.g. /45/).

3. Theoretical Aspects

Theoretical models that account for the SK-relaxation in bcc metals must involve an interaction between dislocations and ISA. In the first model proposed by Köster et al. /11/ diffusion (by reorientation) of ISA in the core of the dislocations, i.e. a Snoek like mechanism, was suggested. Other models proposed by Mura et al. /47/ and Boone and Wert /13/ assumed a cyclical rearrangement of ISA around precipitates.

Other models were suggested for the SK relaxation in iron. Rosenfield /48,49/ interpreted the SK-relaxation in iron as an oxygen Snoek relaxation. Okazaki /50/ proposed that the SK relaxation in iron may be caused by the presence of an fcc structure (induced by deformation) in the bcc lattice. These latter two models can be ruled out clearly from the fact that the SK relaxation occurs in other metals, too. For the other earlier models mentioned above /11,13,47/ the anelastic strain (relaxation strength) should ensue from the reorientation of the ISA. That is the SK peak height should be in the same range of order as that of the Snoek peak. This is in contradiction to the experimental finding that the relaxation strength per solute atom at the dislocations contributing to the SK-relaxation may be about one order of magnitude higher than that for the Snoek peak.

Schöck /7/ first proposed that the SK relaxation should be based on the motion of dislocations, i.e. the species responsible for energy dissipation (anelastic strain) should not be the ISA's but the moving dislocations. According to this model (see below) the SK relaxation should be caused by stress induced motion of dislocation segments (strings), dragging the ISA which become mobile at temperatures higher than the Snoek peak. That is the strength of the SK relaxation should be determined by dislocation movement and can thus considerably exceed that of the Snoek relaxation. Schöck's dislocation-dragging-model has so far found wide acceptance since it could account for most experimental facts.

Recently Seeger /51/ developed a more refined model by taking into account the structure of the dislocations in bcc metals. He proposed that the SK relaxation should be caused by formation of kink pairs in screw dislocations in the presence of ISA. Hirth /52/ proposed a similar model for hydrogen in α -iron. A recent view-point-set (published in Scripta Metallurgica) /53-57/ enlightens the positions of the different authors involved in the development of theoretical models for the SK relaxation in connection with historical surveys.

3.1 String model of the SK relaxation

The dislocations are surrounded by an atmosphere of ISA as shown in Fig. 10a and thus locked at low enough temperatures (i.e. as long as the ISA are immobile). At high enough temperatures (substantially higher than that of the Snoek peak the dislocations may bow out between

immobile locking points under influence of the applied stress by dragging of the now mobile ISA (Fig. 10b). In the dislocation string model this dragging may be introduced by a viscous damping term for the dislocation movement in which enters the concentration of the ISA at the dislocation and their diffusion coefficients. Anelastic relaxation, i.e. mechanical loss, results from this hindered dislocation movement. We discuss this model briefly:

Fig. 10: Models of the SK relaxation. a) position of dislocation without stress. b) string model, c) kink pair formation in $a/2 \langle 111 \rangle$ screw dislocation (kink model).

The loss angle, Q^{-1} , for a simple Debye equation is

$$Q^{-1} = \Delta \omega \tau / (1 + (\omega \tau)^2) \quad (1)$$

with $\omega = 2\pi f$ (f = measuring frequency; An IF peak occurs at $(\omega \tau) = 1$ with the height

$$Q_m^{-1} = \Delta / 2, \quad (1a)$$

from which the relaxation strength Δ can be determined.

The temperature dependence of the relaxation time τ is usually determined by an Arrhenius equation

$$\tau = \tau_0 \exp (H^{SK}/kT) \quad (2)$$

(H^{SK} = activation enthalpy for the SK relaxation, τ_0 = pre-exponential factor). The IF peak temperature T_m results from (2) with $\tau = 1/\omega = \text{const.}$

$$T_m = \frac{H}{k} \ln (\tau_0 \omega) \quad (2a)$$

Schöck /7,55/ derived the following equations for the relaxation strength Δ and the relaxation time τ for the dislocation string model of the SK relaxation:

$$\Delta = \beta \bar{L}^2 \quad (3)$$

(where $\bar{L}$ is the mean length of the dislocation segments involved and $\bar{L}$ is the dislocation density, i.e. their total length per unit volume; β is a numerical factor in the range of 10^{-2} to 10^{-1}) and

$$\tau = \alpha \frac{kT c_d \bar{L}^2}{Gb^3 D'} \quad (4)$$

(α is a numerical parameter of value about unity). In (4) b denotes the Burgers vector, G the shear modulus, c_d the ISA concentration at the dislocations and D' their diffusion coefficient (near the dislocation, "...").

In Schöck's model the main temperature dependence in (4) results from the diffusion coefficient of the ISA

$$D' = D_0 \exp (H^{M'}/kT) \quad (5)$$

where $H^{M'}$ is the migration enthalpy of the ISA near the dislocation. In the undisturbed lattice $H^{M'} = H^S$ (activation enthalpy for the Snoek relaxation).

3.2 Kink model of the SK-relaxation

The kink model introduced by Seeger /51/, takes into the account the specific structure of dislocations in bcc metals, whereas the string model from 3.1 is a more phenomenological one. Screw dislocations with $b = a/2 \langle 111 \rangle$ (a = lattice parameter) are highly immobile (- in contrast to edge dislocations). In IF (anelastic) experiments the bowing out of dislocations under stress is determined by nucleation of kink pairs and their lateral movement to the immobile anchoring points. In pure metals, and for screw dislocations, this mechanism is responsible according to Seeger and Sesták /58/, for the γ -peak (see e.g. /1,2,3, 59/). In presence of ISA, as shown schematically in Fig. 10c, kink movement is strongly impeded by dragging of the ISA. This basic model was proposed by Seeger /51/ for the SK-relaxation in bcc metals.

Seeger /51/ showed that within this kink treatment for moving screw dislocations essentially the same equations for Δ and τ as for Schöck's simpler string model (eqs. 3 and 4) follow. An essential result of the

"kink model" enters into the expression for the activation enthalpy /51/:

$$H^{SK} = 2 H^k + H^{M'}, \quad (6)$$

where $2 H^k$ is the activation enthalpy for formation of a kink pair. Recently Seeger /60/ presented a more refined treatment of the kink model for the SK relaxation. He showed that there may be interference of kink pairs generated under (oscillating) stress with kinks already present in thermal equilibrium. The (linear) density of kinks of one sign in thermal equilibrium on dislocation lines is ρ_k^{eq} . Then the mean distance between kinks present in thermal equilibrium (one sign), x_k , is (/56/):

$$x_k = 1 / \rho_k^{eq} \quad (7)$$

The relaxation time for kink pair formation under stress in presence of an interaction with kinks in thermal equilibrium has been calculated by Seeger /60,30/

$$\tau = \frac{kT}{2a^2 \gamma_d} \cdot \frac{L}{(\rho_k^{eq})^2 \cdot D_k} (1 + \rho_k^{eq} L/2). \quad (8)$$

Here the additional parameter γ_d is the line tension of the dislocations. A detailed discussion of eq. (8) (see /30,60/) shows that, as a consequence, different temperature dependencies of τ (i.e. activation enthalpies) may result. For this the mean distance of kinks, x_k (eq.7), has to be compared with the dislocation loop length L between immobile anchoring points. For low density of thermally produced kinks (low temperatures), i.e. $x_k (=1/\rho_k^{eq}) \gg L$ the relaxation time is

$$\tau \propto L (kT)^2 \exp(2 H_k/kT) / D_k, \quad (9a)$$

(D_k = kink diffusivity), and for higher density of kinks $x_k \ll L$ (high temperatures)

$$\tau \propto L^2 (kT)^{3/2} \exp(H_k/kT) / D_k. \quad (9b)$$

A comparison of eqs. (9a) and (9b) shows that dependent on the criterion $x_k \gg L$ or $x_k \ll L$ (low or high temperatures) the relaxation time τ exhibits different dependencies on loop length (L or L^2) and (more drastically) is determined by different activation enthalpies. ($2 H_k$ or H_k).

This may be understood in a simplified model: The relaxation time for bowing out of dislocations between immobile anchoring points may be expressed as

$$\tau \approx A / (L v_d) \quad (10)$$

(A = area swept out, v_d = mean dislocation velocity). Since $A \propto L^3$ (see e.g. /4,61/) τ may be written as

$$\tau \propto L^2 / v_d \quad (11)$$

The difference between (9a) and (9b) may be seen with (11) as follows:

(a) low temperatures: the density of thermally produced kinks (kink pairs) is low, i.e. their average distance exceeds that of the loop length ($x_k \gg L$). There is no interference of stress generated kinks with thermally produced ones. Thus the dislocation velocity is controlled by the maximum sideward distance a newly generated kink pair can extend. This is L , the (mean) distance between the anchoring points, where the kinks pile up (see Fig. 10c). This gives $v_d \propto L$ and with eq. (11) to $\tau \propto L$. The activation enthalpy is that for kink pair

nucleation, $2 H_k$.

(b) high temperatures: for the opposite case, $x_k \ll L$, there exist on the average one or more kink pairs in thermal equilibrium per dislocation loop length L . This leads to an inference of the kink pairs produced under (oscillating) stress with kinks present in thermal equilibrium and thus to recombination processes: The mean distance travelled by kinks, is reduced to x_k (and becomes temperature dependent). The dislocation velocity is $v_d \propto x_k$. The relaxation time τ , with eq.(11), is $\tau \propto L^2/x_k$. The activation enthalpy is reduced to H_k (since the density of kink sinks increases with T) for $x_k \ll L$.

The transition from case (a) ($x_k \gg L$) to case (b) ($x_k \ll L$) occurs according to Seeger et al /30/ for $x_k = L/2$ and for a value of

$$\hat{\tau} = kT(x_k)^3 / a^2 \gamma_d D_k . \quad (12)$$

The presence of mobile ISA in the neighbourhood of a dislocation leads to an interference by the strain field surrounding the kink. This results in an increase of the kink viscosity or in a dragging force on the moving kink, which may be expressed according to Seeger /51,60/ in terms of the kink diffusivity D_k which enters into eqs. (8) and (9) as

$$D_k \propto \frac{1}{c_d} \exp \left(\frac{-H^S}{kT} \right) .^{+)} \quad (13)$$

With respect to c_d in eq.(13) one has to distinguish between low and high ISA concentrations (for details see /30/, /56/, /60/). Low c_d may lead to a temperature dependent distribution of ISA between dislocation sites (c_d) and bulk sites (c_b) according to

$$c_d = c_b \exp (H^B/kT) \quad (14)$$

By this an additional term containing a binding energy H^B enters into (14) and thus as an additional temperature dependence into D_k (eq.13). For high ISA concentration the dislocation sites may be saturated and no temperature dependent term appears in (13).

From the preceding description it follows that four limiting cases may be derived according to Seeger /56/ for the effective activation enthalpies $H_{eff} = d \ln \tau / d(1/kT)$ of the SK relaxation. The different cases are obtained from equations (9a) for low temperatures or (9b) for high temperatures together with eq. (13) (if there enters a binding enthalpy or not). The results are listed in Table 1. The kT -terms are a consequence of the pre-exponential terms in eqs. (9a) and (9b). The difference in H_{eff} between the case of low and high temperatures is $H_{eff}^{(l)} - H_{eff}^{(h)} = H_k - \frac{1}{2} kT \approx H_k$. This will be tested in sect. 4 with respect to experimental results.

	c_d small	c_d high
$x_k \gg L$ (low temperatures)	$2H_k + H^S + H^B - 2kT$	$2H_k + H^S - 2kT$
$x_k \ll L$ (high temperatures)	$H_k + H^S + H^B - 3/2 kT$	$H_k + H^S - 3/2 kT$

Table 1: Effective activation enthalpies H_{eff} for the SK relaxation acc. to Seeger /56/.

⁺) In eq. (13) a term $\exp(-H_k^M/kT)$ with H_k^M = kink migration enthalpy can be omitted since H_k^M for bcc metals is in the range of 10^{-2} eV (see e.g. /30/), i.e. $H_k^M \ll kT$ for usual temperatures.

The temperature dependence of the relaxation time for the low and high temperature case according to eqs. (9a), (9b), and (13) (assuming $H_B = 0$, i.e. c_d high enough) is shown schematically in Fig. 11 in an Arrhenius diagram for two dislocation loop lengths L_1 and $L_2 > L_1$ (this will be discussed below). The break for the transition from low temperatures (right in $1/T$ scale) to high temperatures (left) is controlled by eq. (12) (see 4.2.2).

4. Discussion of Experimental Results on the SK relaxation

In the preceding survey on theoretical models for the SK-relaxation it was shown that the strength of the relaxation as given by the two models is the same, since both models involve the concept of bowing out of dislocations fixed at immobile nodes (see e.g. /61/. Thus in both models the specific nature of the SK-relaxation, the interaction with foreign atoms - and the statements from different models - enters into the relations for the relaxation time.

4.1 Relaxation strength

4.1.1 Temperature dependence. - According to the dislocation models for the SK-relaxation, the relaxation strength Δ should be independent of temperature (eq. 3). For a deformed Nb-O (100 at.ppm) monocrystal the temperature dependence of the high temperature relaxation process (2) has been studied in /30/ by a combination of IF and mechanical after-effect experiments. The results for three different degrees of plastic deformation are shown in Fig. 12. As can be seen from Fig. 12 Δ is indeed independent of temperature. As far as we know this has been verified for the first time for a deformation induced peak. This gives strong support for the dislocation model and clearly rules out other models /11, 47-50/, in which a Snoek type reorientation — for which $\Delta \propto 1/T$ is expected — was assumed as basic mechanism of the SK relaxation.

4.1.2 Dependence on amount of plastic deformation. - According to eq. (3) it is expected that $\Delta \propto \bar{\Lambda} \cdot \bar{L}^2$, i.e. the product of the dislocation density $\bar{\Lambda}$ and the square of the (mean) loop length ($\bar{L}^2$) enters into the expression for Δ . One expects that Δ increases with increasing amount of plastic deformation due to an increase of the dislocation density (unless $\bar{L}$ decreases very drastically with increasing deformation). This is seen in most experiments dealing with the influence of degree of deformation on the SK relaxation (and considered as a criterion that indeed a SK-process occurs). Detailed investigations of the influence of degree of deformation were performed e.g. on Fe (/11/, /18/, /21/-/23/, /37/), Ta (/12, 40/) and Nb (/14, 30/). Examples are reproduced in Fig. 2 and Fig. 5 for Fe and Fig. 7 for Nb. These clearly show that the deformation induced peaks increase with amount of plastic deformation (in Fig. 5 and 7 for both peaks (1) and (2)).

4.2 Relaxation time

4.2.1 Pre-exponential factor τ_0 . - The pre-exponential factor for both, the string model (eq. 4) and the kink model (eqs. 9a, 9b) depends on the state of the sample. The main parameters which enter into eqs. (4) or (9) are the loop length L (in the string model L^2 , in the kink model L or L^2) and the concentration of ISA at the dislocations, c_d . According to eq. (2a) the IF peak temperature T_m directly reflects variations in τ_0 ($T_m \propto \ln \tau_0$).

Fig. 11: Schematic Arrhenius diagram ($\ln \tau$ vs. $1/kT$) for the kink model and two discrete dislocation segment lengths L_1 and L_2 [23/.

Fig. 12: Temperature dependence of the relaxation strength Δ for Nb-O and three degrees of plastic deformation (a, b, c; for details see [30/).

(a) Influence of plastic deformation.— With increasing plastic deformation the loop length L , i.e. the distance between nodes in the dislocation network should decrease. Thus, also T_m should decrease with plastic deformation. This was found in many publications on the SK relaxation. Fig. 5, e.g. shows, for Fe-N, a slight decrease of T_m with increasing amount of deformation for both peaks (1) and (2). A similar behaviour is seen in Fig. 7 for Nb-O. This result is in good qualitative agreement with the dislocation models.

(b) Influence of annealing.— Annealing treatments after plastic deformation are expected to again increase the loop lengths. This is e.g. demonstrated in Fig. 5 for the example of Fe-N.

(c) Peak broadening.— After plastic deformation the dislocation network usually consists of a distribution with different loop lengths L peaking at a mean value $\bar{L}$. A distribution in L leads to a distribution in τ , i.e. a broadening of the SK peaks. An exponential distribution in L values was already introduced by Schöck [7/]. In most experiments on the SK relaxation it is reported that the SK peaks are essentially broader than a simple Debye peak. Only a few experiments are known in which a quantitative evaluation of the peak shape was tried. Usually a log-normal distribution which may be characterized (according to Nowick and Berry [4/]) by a distribution parameter, β , was applied. Buchmann and Kennedy [20/] obtained $\beta \approx 3$ for Fe-N, Magalas et al. [21/] $\beta \approx 3.5$ for Fe-C. In detailed studies on Nb-O [30/], by using IF and mechanical after-effect experiments, a temperature independent $\beta \approx 3.5$ was obtained. This strongly indicates, that, as analysed in [30/], the broadening of the IF peaks indeed results from a loop length distribution and not, as may also be discussed, from a distribution in activation enthalpies.

4.2.2 Activation enthalpy H^{SK} .— The activation enthalpy of the SK-relaxation is the parameter which most specifically reflects the physical nature of the relaxation process in the different models (string or kink). The experimental fact that H^{SK} is considerably higher than H^S — the difference $H^{SK} - H^S$ is in the range of 0.5 eV — is interpreted in both models in a different manner:

(i) Within the frame of the string model two explanations were suggested. Schöck and Mondino /12/ argued that for diffusion near the dislocation core interstitial locations with different tetragonalities may lead to an extra energy barrier. As pointed out by Seeger /49/ this explanation cannot hold for $a/2 \langle 111 \rangle$ screw dislocations in bcc metals since the stress field of the dislocations has trigonal symmetry so that the various sites of $\langle 100 \rangle$ oriented ISA dipoles are energetically equivalent. Another objection against ISA with tetragonal strain fields is derived from the experimental fact that pronounced SK peaks of hydrogen - the tetragonality of which is extremely small - are observed in α -Fe and group V transition metals. The same difficulty arises in explaining recent experiments in which a SK peak was reported for fcc Ni containing carbon /62/ which occupies isotropic interstitial sites.

(ii) Within the kink model as outlined in section 3.2 and summarized in Tab.1 the difference $H^{SK} - H^S$ may vary within wide limits. The minimum value is H_k , the maximum value $2H_k + H^B$. An extra term of a binding energy H^B was already proposed by De Batist /5/ who suggested that the ISA are partitioned between lattice (bulk) sites and dislocation sites according to eq. (14).

For a quantitative discussion of the experimental activation enthalpies published in literature within the theoretical framework of the kink model outlined above, it is evident that the internal state of the samples (C_d , degree and mode of deformation) must be known in detail. Only for special cases as listed in Table 1, may theoretical equations directly be applied - as was shown for Nb-O /30/.

Additional information may be obtained: (i) From IF experiments on deformed pure bcc metals, i.e. data on the γ -peak (α -Fe /63-65/, Nb /66/, Ta /40/), from which values for the kink pair formation enthalpies may be derived.

(ii) From measurements of the temperature dependence of the critical resolved shear stress in bcc metals for which, as shown by Seeger/67/ the theory of kink pair formation is also applicable. For a quantitative discussion of the experimental activation enthalpies of the SK relaxation in Nb-O and Ta-O, together with (i) and (ii), see /30/, and for Fe see /51/.

4.3 Two Snoek-Köster Peaks

From experimental results on deformed Fe, Nb and Ta as represented in the Arrhenius diagrams of Figs. 3, 6 and 8, it can be concluded that there exist two deformation-induced peaks designated as peaks (1) and (2). The occurrence of two SK peaks may be explained by the dislocation model by assuming that two drastically different distributions of loop lengths $\bar{L}$ exist, which peak at two values L_1 and L_2 (see also discussion in /23/) and that differ at least one order of magnitude from each other.

This behaviour is shown schematically in Fig. 11 in an Arrhenius type diagram ($\ln \tau$ vs. $1/kT$ /23/) for two discrete loop lengths L_1 and $L_2 > L_1$. At the transition from the low temperature case ($x_k \gg L$, $\tau \propto L^1$, eq. 9a) to the high temperature case ($x_k \ll L$, $\tau \propto L^2$, eq. 9b), at $\hat{c}$ (eq. 12), the slope of the lines, H^{SK} , is reduced by H_k . Fig. 11 shows that in IF-experiments which are carried out at constant $\tau = 1/2 \pi f$ (dashed line) two peaks may result under these conditions. Within the $\ln \tau - 1/T$ range, marked by dotted lines, the low temperature peak is characterized by a higher slope (activation enthalpy) than the high temperature peak. Apparently this is the case for Nb-O /30/.

The origin of two L distributions is not yet clear. In /30/ it was suggested that small precipitates may subdivide long dislocations into

smaller segments which would cause the low temperature peak (1).

An alternative interpretation was offered in /23/. In deformed metals there may exist wide regions with long dislocation segments and narrow regions with densely packed, much shorter segments. Thus two groups of lengths, L_1 and L_2 , may result and thus lead to two, well separated SK peaks. Another specific prediction from the kink model is that for two SK peaks the activation enthalpy for lower temperatures may exceed that for higher temperatures (compare Table 1); the difference should be equal to $H_K - 1/2$ kT. This prediction was verified for Nb-O /30/ (see sect. 2.2.1). According to /30/ the activation enthalpy for the low temperature peak ($x_k \gg L$) is $H_{\text{eff}}^{\text{SK}}(1) = (2.00 \pm 0.18)$ eV, and for the high temperature peak ($x_k \ll L$) $H_{\text{eff}}^{\text{SK}}(2) = (1.68 \pm 0.015)$ eV and thus $H_K = H_{\text{eff}}^{\text{SK}}(1) - H_{\text{eff}}^{\text{SK}}(2) + 1/2$ kT = 0.32 eV + 0.03 eV = 0.35 eV is obtained. This H_K value is (within experimental error) in good agreement with de Lima's result /66/ derived from the γ -peak in pure Nb ($H_K = 0.33$ eV).

4.4 Miscellaneous and open questions

(i) Different behaviour of Fe-C and Fe-N. — Originally it was claimed /18/ that C does not contribute to the SK-relaxation in Fe, but then Kamber et.al. /16/ demonstrated that the SK-relaxation of C in α -Fe exists but only after annealing to higher temperatures than the SK peak temperature (see 2.1). An explanation for this apparently different behaviour was given by Mondino and Seeger /68/. Cold-working generates both dislocations and vacancies. At heating up in IF experiments, complexes of C or N with vacancies are formed (migration of C or N at $T \approx 350$ K). IF and positron annihilation experiments on neutron irradiated iron doped with C or N demonstrated that the dissociation of these complexes - which is a pre-requisite for appearance of the SK-peak - occurs for Fe-N at about 500 K and Fe-C at 600 K /69-71/. This explains why the SK-N(1) peak appears in the first heating up curve, whereas the SK-C(1) peak appears only after heating to higher temperatures (≥ 600 K) in a second run. The influence of vacancy-ISA-interaction on the development of the SK relaxation should not be confined to Fe but should also dominate in other bcc metals if the dissociation of vacancy-ISA complexes occurs at higher temperatures than the SK peak temperature. Considering group-V transition metals — non metal systems, Nb-N may behave similarly as Fe-C, Ta-O and Nb-O like Fe-N /68/.

Another explanation which additionally may play a rôle was advanced by Fast /8/. Carbon in α -Fe has a lower solubility than nitrogen and may precipitate as carbide near dislocations and/or grain boundaries /72/. Heating to higher temperatures dissolves the precipitates and develops the SK peak. This mechanism may dominate for low degrees of cold-work (low vacancy concentration).

(ii) "Undeformed" metals. — Dahlstrom et.al. /73/ showed that an SK peak appeared in quench-aged Nb-N without external deformation (as in /13/). Transmission electron microscopy studies showed that dislocation loops and tangles of significant density were created around coarsened (quench-aged) precipitates, which apparently are responsible for the SK-relaxation. Similar conditions may predominate in Fe-(C+N) alloys /74/ which showed SK(1)-peaks after rapid quenching.

The SK(2) peak was observed in /23/ in undeformed Fe-N monocrystals (see Fig. 5, curve 1) and in deformed high purity iron. By this an intrinsic relaxation mechanism for this high temperature SK(2)-peak may not be ruled out completely. Other relaxation mechanisms occurring at high temperatures ($\approx 0.5 T_{\text{melt}}$) may be discussed within this context (for a recent review see /75/). To these belong mechanisms such as climb of dislocations by movement of jogs and/or participation of vacancies in polygonised dislocation arrangements /76-78/. These structures may also be present in monocrystals. By using magnetic after-effect measurements

Kuke et.al. /79,80/ found after slight deformation of pure Fe and Fe-C a peak at ≈ 620 K (and another, smaller one at 370-420 K) for $\tau = 190$ s. Their 620 K peak may be identified as SK-2, the low temperature peak as SK-1 (their data fit well in the Arrhenius diagram of Fe in Fig. 3). Kuke et.al. suggested for their 620 K peak a relaxation process involving non conservative movements of jogs, i.e. an intrinsic mechanism without participation of ISA. However, the correlation of the high temperature peak (2) in deformed Fe, Nb and Ta to a relaxation of the SK-type seems to us at the moment more straight forward.

(iii) Transient effects, "pure" metals. — Studies of the SK peak as a function of ISA-concentration (e.g. Fe-N /18/) point to the existence of an SK-peak at (extrapolated) zero concentration, i.e. in samples which exhibit no detectable Snoek peak.

The range of extremely low concentrations is not covered by SK-theories presented in this paper. For low ISA-concentrations, c_d , the dislocation dragging model for the SK-relaxation is no longer applicable and break away processes have to be considered /51/. As pointed out by Seeger a gradual transition from the SK to the γ -relaxation-process has to be expected. This transition was shown by Oytana et.al. /31/ for Nb-O (higher O-concentrations). Activation enthalpy and IF peak temperature are expected to be higher than that of the γ -relaxation but less than that of the normal SK relaxation (c_d high enough) for kink pair formation and break away processes. To this category apparently belong transition effects published e.g. in experiments on Fe-C (N) /81-85/.

These transient effects should be distinguished from the enhancement and often asymmetric broadening of the Snoek peak as reported e.g. for Fe-C, N /84-87, 23/, or Nb-O /30/ (see Fig. 7). This dislocation enhanced Snoek effect is attributed /30/ to dislocation lines of nonscrew type with small Peierls barriers (a sort of "SK relaxation of edge dislocations").

(iv) Type of dislocations involved. — A specific prediction of the kink model for the SK-relaxation of heavy ISA in bcc metals is that $a/2 \langle 111 \rangle$ screw dislocations are involved and that the relaxation strength of the SK-relaxation should be the same as that of the γ -relaxation for a given dislocation pattern. These two predictions could not be verified up to now clearly.

Heights of the SK-(1) peaks reported for Fe and group Va - transition metals are in the same order of magnitude as those reported for the γ -peaks in these metals. The influence of deformation at lower temperatures — which should lead to a stronger influence of screw dislocations — was studied for Fe-N /22/ and Fe-C /21/. Both experiments were restricted to temperatures below ≈ 700 K, i.e. only peak SK-(1) was studied so that the conclusions (disagreement with the kink model) drawn by the authors /21/ should be reflected upon once more in the light of the new experimental finding of two SK-peaks.

Without doubt further experimental and theoretical work is necessary to get a deeper insight in the mechanism of the Snoek-Köster relaxation.

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