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Study of non-linear effects related to the Snoek–Köster relaxation in Nb

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Abstract

The variation of mechanical loss (internal friction) with the amplitude of oscillation, ϵ, was studied in Nb–O with various oxygen contents (100–1200 at.ppm) by applying an inverted torsion pendulum ($f \simeq 3$ Hz). The Snoek–Köster maximum was analysed with respect to various non-linear effects: amplitude-dependent damping, peak temperature shift, peak width and asymmetry. The asymmetry was judged from the low- and high-temperature halfwidth of the maximum, r_{LT} and r_{HT}. In all specimens we observe an increase in the peak heights and r_{HT} with increasing ϵ, whereas the peak temperatures and r_{LT} decrease with ϵ. The non-linear effects are discussed with respect to the theory of the SK relaxation involving kink-pair generation in screw dislocations in the presence of interstitial foreign atoms.

1. Introduction

Mechanical loss (internal friction) experiments are well suited to the study of intrinsic properties of dislocations [1, 2]. Usually, mechanical loss spectra resulting from dislocation movement are observed as amplitude-independent damping at small oscillation amplitudes. Observation of amplitude-dependent damping (ADD) dates back to the early work of Köhler [3] and Granato and Lücke [4], who proposed a model based on breakaway of dislocations from pinning points. Amplitude-dependent damping represents non-linear behaviour. This is contrary to ideal (linear) anelasticity, for which we expect that doubling of the applied stress leads to doubling of the resulting strain.

As pointed out by Alefeld *et al.* [5], the amount of information on dislocation relaxations may be increased considerably by extending the observations to non-linear effects. For this purpose, mechanical loss (Q^{-1}) and modulus (frequency) are measured as a function of oscillation amplitude. Non-linear effects may be expected, *e.g.* if dislocation movement proceeds via Peierls–Nabarro potentials. One example is the Bordoni relaxation in f.c.c. metals, which, according to the theory of Seeger [6], is due to the thermally activated generation of kink pairs in screw dislocations.

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Concerning b.c.c. metals, it is generally accepted now that the γ-peak is due to formation of kink pairs in screw dislocations. In the presence of interstitial solute atoms it transforms into the Snoek–Köster relaxation [7, 8]. For both cases, non-linear effects may be expected. Recently [9] the authors reported on amplitude-dependent damping in a Nb monocrystal doped with 400 at.ppm oxygen in the temperature of the Snoek–Köster relaxation. This paper presents a more systematic examination of non-linear effects in niobium with various oxygen contents.

2. Experimental

2.1. Samples

The samples were prepared from ultrapure Nb, which after decarburizing and degassing in ultra-high vacuum had a resistivity ratio $\rho_{300\,K}/\rho_{4.2\,K}$ of about 6000. Oxygen contents between 100 and 1200 at.ppm were introduced by heating in pure O_2 atmosphere (10^{-7}–10^{-8} mbar) at 2200–2400 K. The samples are listed in Table 1. Finally, the specimens were coated with a 1 μm thick layer of Ti to prevent oxygen pick-up during internal friction measurements at higher temperatures [10]. For details of sample preparation see [11].

2.2. Measurements

The measuring apparatus was an inverted torsion pendulum operating in the frequency range 1–15 Hz.

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TABLE 1. List of specimens and values of $\bar{s}$ and H_{SK} (after 1.5% tensile + 1% torsional deformation)

Sample	Oxygen content [at.ppm]	$\bar{s}=\Delta Q^{-1}/\Delta\epsilon$ at SK peak	H_{SK} (eV)
P polycrystal	300	190	1.95±0.1
E1 monocrystal ⟨110⟩	400	530	1.57±0.04
E2 monocrystal ⟨110⟩	1200	120	1.76±0.04
E3 monocrystal medium orient. ($\chi=8°$, $\lambda=41°$)	100	73	1.90±0.05

The freely decaying torsional amplitudes A_n ($n=1, ..., N$) were measured at constant temperature T with an automatic data-acquisition system (HP series 200 computer with HP 3437 DVM). In a first step, polynomials were fitted to the data, ln A_n *vs.* n, representing the decaying oscillations. The internal friction as a function of amplitude, Q^{-1} (A), was determined by fitting polynomials to the inverse function $n(\ln A)$ according to

$$Q^{-1}(A) = -\frac{1}{\pi}\left[\sum_{i=1}^{p} ic_i(\ln A)^{i-1}\right]^{-1} \quad (1)$$

Best fits were obtained with polynomials of degree $p \leqslant 3$. The results are depicted as Q^{-1} *vs.* shear strain ϵ (ϵ = const. A). As a criterion for the amplitude-dependent damping we introduce the slope of the $Q^{-1}(\epsilon)$ curves, $s = dQ^{-1}/d\epsilon$. This may be replaced in a restricted range of ϵ by the mean value $\bar{s}=\Delta Q^{-1}/\Delta\epsilon$.

3. Results

Measurements of amplitude-dependent damping for polycrystal P (doped with 300 at.ppm O) after plastic deformation (1.5% tensile + 1% torsion at 300 K) are shown in Fig. 1. $Q^{-1}(\epsilon)$ was determined from the decreasing oscillation amplitudes (f ≃ 2.5 Hz) in the range of 8 to 2×10^{-6} with eqn. (1). Figure 1 shows Q^{-1} *vs.* ϵ for various temperatures as indicated. Values of the parameter $\bar{s}=\Delta Q^{-1}/\Delta\epsilon$ are marked on the right-hand side for a mean oscillation amplitude of $\epsilon_m = 5\times10^{-6}$. The $Q^{-1}(\epsilon)$ curves for the temperatures of the O-Snoek peak (434 K) and Snoek–Köster peak (697 K) are depicted as dashed lines.

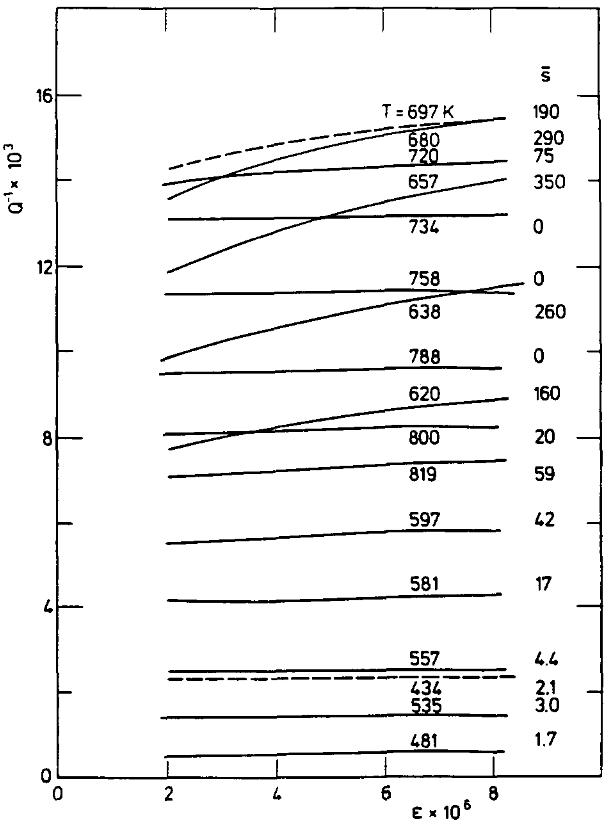

Fig. 1. Q^{-1} *vs.* oscillation amplitude ϵ of specimen P (Nb–300 at.ppm O) after plastic deformation (1.5% tensile + 1% torsion) for various temperatures.

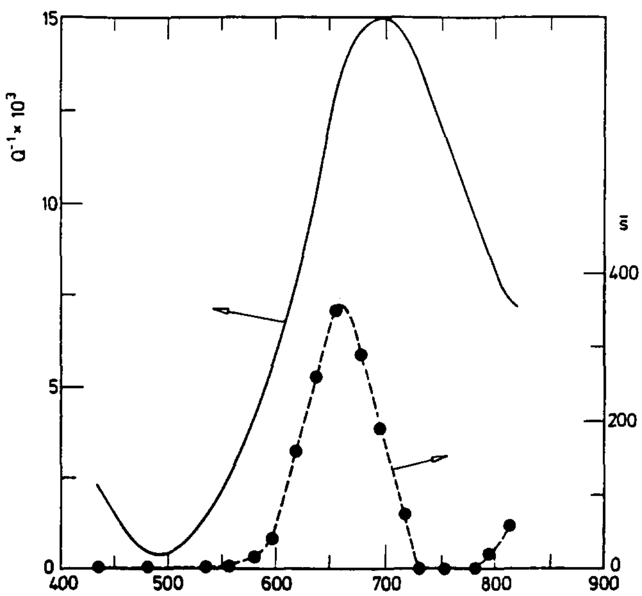

Fig. 2. Parameter $\bar{s}=\Delta Q^{-1}/\Delta\epsilon$ and Q^{-1} *vs.* temperature for a mean oscillation amplitude ($\epsilon=5\times10^{-6}$) for specimen P.

Figure 2 shows the variation of the parameter $\bar{s}$ with temperature for specimen P. Additionally we show the Q^{-1} *vs.* T curve for a mean oscillation amplitude ($\epsilon_m = 5\times10^{-6}$) indicating the SK-2 peak. The SK-1 peak (expected at about 550 K [10]) is absent, as the specimen was pre-annealed to about 850 K before taking the

$Q^{-1}(\epsilon)$ curves in the second heating-up (see. *e.g.* [8]). As can be seen from Fig. 2, the parameter $\bar{s}$ characterizing amplitude-dependent damping increases in the temperature range of the SK-2 maximum and has its maximum value ($\bar{s}=350$) at ≈ 657 K, close to the SK-peak temperature. Measurements of $Q^{-1}(\epsilon)$ for the other specimens (monocrystals E1, E2, E3; see Table 1) exhibit a similar behaviour. The parameter $\bar{s}$ has its maximum in the range of the SK maximum and is negligibly small at lower and higher temperatures. This clearly indicates that amplitude-dependent damping, *i.e.* non-linear behaviour, is closely related to the Snoek–Köster relaxation.

The values of $\bar{s}$ at the temperatures of the SK maximum are included in Table 1 [12]. To obtain further information on the 'non-linear' behaviour of the SK maximum we analysed the SK maxima, after background subtraction, with respect to peak height ($Q^{-1}{}_p$), peak temperature (T_p) and symmetry. As an empirical parameter for symmetry we determined the half-widths of the maximum on the low-temperature side, r_{LT}, and on the high-temperature side, r_{HT}, according to

$$r_{LT}=0.759\,\frac{H}{k}\left[\frac{1}{T_{LT}}-\frac{1}{T_p}\right];$$

$$r_{HT}=0.759\,\frac{H}{k}\left[\frac{1}{T_p}-\frac{1}{T_{HT}}\right] \tag{2}$$

where k is Boltzmann's constant, and T_{LT} and T_{HT} are the half-width temperatures on the low- and high-temperature sides respectively. Values of the activation enthalpy H for the different specimens were determined with the frequency-shift method and are listed in Table 1 (the values are taken from [10] and [13]).

The half-peak widths r_{LT} and r_{HT} are defined in such a way that for an ideal Debye peak we expect $r_{LT}=r_{HT}=1$. Values larger than 1 indicate a broadening of the maximum, *i.e.* a distribution of relaxation times [16]. For linear (amplitude-independent) behaviour as *e.g.* for a Debye peak, all parameters should be independent of the oscillation amplitude ϵ. The results of the peak analysis are shown in Figs. 3–5. Figure 3

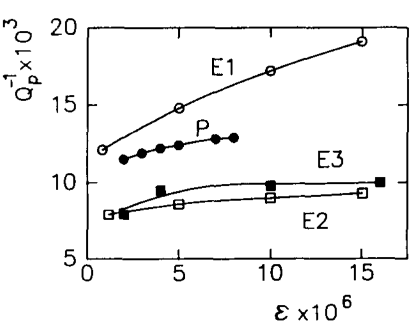

Fig. 3. Variation of SK peak heights $Q^{-1}{}_p$ with oscillation amplitude ϵ for different specimens.

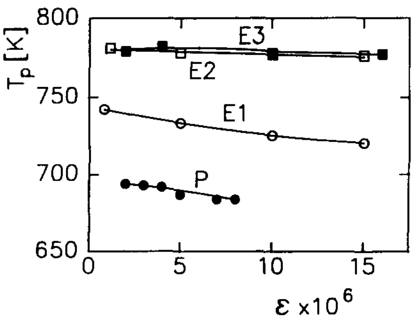

Fig. 4. Variation of SK peak temperatures T_p with oscillation amplitude ϵ.

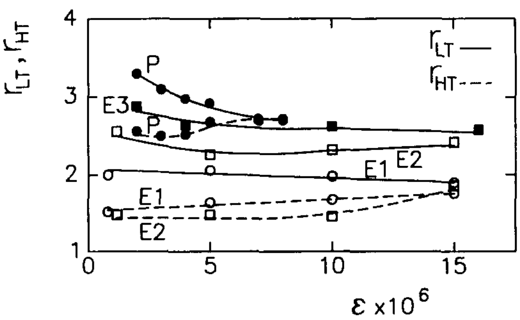

Fig. 5. Variation of peak width parameters r_{LT} and r_{HT} with oscillation amplitude ϵ.

shows that the peak height $Q^{-1}{}_p$ increases with ϵ. The peak position T_p depicted in Fig. 4 is shifted to lower temperatures with increasing ϵ for all specimens. This behaviour was already observed earlier in deformed Nb–O [8]. Figure 5 shows r_{LT} and r_{HT} *vs.* ϵ. For all specimens we observe a more or less pronounced decrease of r_{LT} and an increase of r_{HT} with ϵ. This indicates that the SK maxima exhibit an asymmetrical broadening on their high-temperature side with increasing ϵ.

Discussion and conclusions

As shown in Figs. 2–4, the amplitude-dependent effects are stronger for specimens with lower oxygen contents (especially for specimens P and E1). The different behaviour of r_{HT} and r_{LT} gives information on the change in the symmetry of the peaks with ϵ. Figure 5 shows that, with increasing ϵ, the SK-2 peak becomes broader at the high-temperature side and narrower at the low-temperature side, whereas the total width $(r_{LT}+r_{HT})/2$ increases or decreases with ϵ. Dislocation breakaway can be disregarded as origin of the non-linear behaviour. For a breakaway mechanism we expect a monotonic increase of s with temperature [4]. Such a behaviour was, for example, observed for crystal E1 before deformation. However, a Friedel's plot [14] (log of the amplitude-dependent component of Q^{-1} against strain) does not give a straight line as is predicted in a simple model [14] for thermally assisted breakaway of dislocations. The results for crystal E2 indicate that higher contents of interstitial foreign atoms (IFA) reduce the amplitude-dependent effects. For crystal E3, the different (medium) orientation also leads to a reduction of the non-linear effects. The larger effects in specimen E1 (with lower oxygen content) may be due to longer dislocation loops compared to shorter, more tangled dislocation segments expected in the polycrystalline sample P, or the shorter dislocation segments in the presence of atmospheres of IFA expected in E2 with a high content of oxygen. The decrease of T_p with ϵ (Fig. 4) corresponds to a decrease of the activation enthalpy with increasing ϵ, from which an activation volume may be calculated. For samples P and E1 the activation volume may be estimated to 500–1000 b^3. This is close to, but a little larger than, the quantity expected for kink-pair formation in screw dislocations of pure Nb [9].

As far as we know the non-linear effects reported earlier [9] and in the present work constitute the first indication given in literature about the presence of ADD associated with the mechanism of the SK relaxation involving heavy IFA. There is no model devoted to explain ADD associated to the SK relaxation. Such a model should take into account the following characteristics:

(1) $\bar{s}$ has its maximum value in the range of the SK peak and decreases on its HT side. The SK maximum increases with increasing strain amplitude ϵ.

(2) T_p is shifted to lower temperature with increasing ϵ.

(3) The high-temperature half-peak width increases with increasing ϵ.

(4) The low-temperature half-peak width decreases slightly with increasing ϵ.

(5) Maximum ADD occurs at relatively small IFA concentrations.

Esnouf and Fantozzi [15, 2] presented a modification of the Seeger–Paré model for the Bordoni relaxation in f.c.c. metals, which includes non-linear effects. In their multi-well model [15] the activation enthalpy for nucleation of kink pairs depends only on stress, whereas the annihilation enthalpy is a function of stress, dislocation length and well number. They predicted marked non-linear effects if the vibrating stress is greater than the static one (internal or applied). Their calculations gave an increase of $Q^{-1}{}_m$ with ϵ (up to a peak), a decrease of T_p, and an increase of both r_{LT} and r_{HT} (only slight for r_{LT}) with increasing vibrating stress. The fact that the essential features of the non-linear effects observed for the SK relaxation are similar, even though not equal, to that of the Bordoni peak in f.c.c. metals according to the model of Esnouf and Fantozzi, may lead to the following conclusions:

(1) The interstitial foreign atoms (IFA) play a secondary role for ADD. An elevated concentration of IFA leads, however, to the reduction or suppression of the effects.

(2) The mechanism for ADD can be related to what is the common feature of both relaxation mechanisms: thermally activated generation of kink pairs, in spite of the different structure and mobility of dislocations in b.c.c. and f.c.c. metals.

New refinements have to be introduced into the original models for the SK phenomena to account for the non-linear effects presented and analysed in this paper.

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