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DISLOCATION RELAXATION PEAKS IN MOLYBDENUM

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Abstract.- In high purity molybdenum single crystals measurements of the damping and modulus are reported in the temperature range of the α- and γ-relaxation. Activation volumes of the α-subpeaks were determined from the amplitude dependence of the Internal Friction. The results clearly support the explanation of the α-relaxation in terms of kink pair formation in 71°-dislocations and diffusion of different types of geometrical kinks in screw dislocations. Deformation induced point defects interact with these dislocation segments giving rise to the β-relaxation. Some characteristics of the γ-peak (kink pair formation on screw dislocations) are discussed.

1. Introduction.- Internal Friction (IF or $Q^{-1}$) is far more complex in b.c.c. metals than in f.c.c. metals due to the special core structure of screw dislocations in the b.c.c. lattice /1/ and the high sensitivity for interstitial impurities. In our group the problem has been approached by two complementary sets of experiments. Systematic doping of niobium and tantalum elucidated the influence of hydrogen and oxygen onto the damping spectra /2,3/. The present work focuses on the intrinsic barriers to dislocation motion using high purity molybdenum. The measurement of "secondary features", especially the amplitude dependence of the damping peaks delivers a decisive test of the suggested models /4/.

2. Experimental Procedure.- The experiments have been carried out in a torsion pendulum in the temperature range between 10 K and 700 K. To determine activation parameters the resonant frequency was changed by a factor of 50 in the range between 0.4 Hz and 26 Hz. In different high purity molybdenum single crystals (residual resistance ratio RRR=40000) the influence of the crystallographic orientation of the sample, the amount and temperature of deformation and the aging treatment on IF have been investigated. All damping spectra were recorded as a function of temperature and strain amplitude $A$ ($A$ being between $5 \times 10^{-6}$ and $2 \times 10^{-4}$) using a method of measurement described elsewhere /5/.

3. Experimental Results.- The damping peaks will be discussed in the nomenclature of Chambers /6/ using the symbols $\alpha$, $\beta$, and $\gamma$ according to...
The damping spectra obtained after low temperature deformation (curve 1, \( \varepsilon = 1\% \) at 200K) and after high temperature deformation (curve 2, \( \varepsilon = 1\% \) at 475K) are shown in figure 1. Curve 1 is enhanced by a relative scaling factor of 2.5 to obtain comparable peak heights. It can be seen, that (i) the \( \gamma \)-peak is significantly increased by low temperature deformation, and (ii) the relative heights of the low temperature parts of the \( \alpha \)-relaxation are enhanced by low temperature deformation, whereas the main process at 115 K (\( \alpha_1 \)) is increased by high temperature deformation.

The \( \beta \)-peak appears here as a shoulder of the \( \alpha \)-relaxation located between 110 K and 250 K. This process disappeared after annealing at 600 K (fig.2a, curve 2) and the \( \alpha \)-peak reached a stable configuration showing a pronounced 4-fold substructure. The corresponding modules curves (fig.2b) reveal that the aging treatment does not affect the overall dislocation mobility below room temperature. This confirms that no dislocation pinning by interstitials like nitrogen, oxygen or carbon took place and, therefore, the annealing effects must be caused by deformation induced point defects or by a dislocation rearrangement.

In different b.c.c. metals the \( \alpha \)-relaxation is composed of several processes (8,9,2). Any conclusive interpretation of the single process in terms of the suggested models requires a separation of the subpeaks. However, deconvolution is only useful if it is unique and allows to obtain distinct characteristics of the subpeaks. In figure 3 the decomposed \( \alpha \)-relaxation is shown at two frequencies. The subpeaks are symmetrical in a \( Q^{-1} \) vs. \( 1/T \) plot. This assumption is fulfilled if processes with symmetrically distributed relaxation times contribute to the \( \alpha \)-relaxation /9/.

Figure 4 shows the \( \alpha \)-relaxation at different strain amplitudes. The observed amplitude dependence belongs just to the relaxation processes observed at low amplitudes. Even the \( \alpha \)-peak substructure is reflected in the amplitude dependent part of the damping (fig.4, below). The subpeak \( \alpha_1 \) is more separated from \( \alpha_2 \) at higher amplitudes (compare fig.4 above and below).

Activation volumes are calculated from the temperature shift of each subpeak through a variation of the applied strain amplitude. Details of the evaluation procedure and further results will be published. The activation parameters for the \( \alpha \)-relaxation in the [100]-orientated crystal are presented in table 1 using a Schmid factor for the torsional stress of 0.58.

The \( \gamma \)-relaxation has been associated with the basic mechanism controlling the macroscopic flow in b.c.c. metals at low temperatures /6,7/. Because of the high purity of our crystals we were able to observe the
Fig. 1: Influence of the deformation temperature on the damping spectrum. Curve 1 is enhanced by a relative scaling factor of 2.5.

Crystal orientation: [111]

- Frequency \( f = 25 \text{ Hz} \)
- Curve 1: \( \varepsilon = 1\% \) tension at 200 K + 0.2\% torsion at 290 K
- Curve 2: \( \varepsilon = 1\% \) tension at 475 K + 0.2\% torsion at 475 K

Fig. 2a: Annealing behaviour after low temperature deformation.

1: as deformed, last deformation 0.2\% torsion
2: annealed for about 15 min at 600 K

Fig. 2b: Modulus curves \((G-f^2)\) corresponding to the damping curves of fig. 2a.
Fig. 3: (above to the left)
Decomposition of the α-peak into 4 subpeaks at two measurement frequencies.
above: \( f = 18.5 \) Hz
below: \( f = 0.4 \) Hz

Fig. 4: Amplitude dependence of the α-relaxation, sample annealed 1/2 hour at 700 K after low temperature deformation.
crystal orientation: [100]
frequency \( f = 14 \) Hz
the strain amplitudes increase by an amount of \( \Delta A = 2.9 \times 10^{-5} \) respectively
\( A = 6.8 \times 10^{-6} \) for curve 7
\( A = 1.8 \times 10^{-4} \) for curve 1

below: Amplitude dependent part of the α-relaxation (difference between curve 5 and 1 from above).

Fig. 5: (above to the right)
The γ-relaxation compared to a single Debye peak.
.. measurement points
-- damping background subtracted
--- Debye peak
Table 1: Activation parameters for the α-subpeaks. The applied stress amplitudes $\sigma_a$ are given in units of the shear modulus $G$, the activation volumes $v$ in units of the Burgers vector $b$.

<table>
<thead>
<tr>
<th>$\sigma_a$</th>
<th>$H[\text{eV}]$</th>
<th>$f_0[1/\text{s}]$</th>
<th>$v$ for $\sigma_a = 5 \times 10^{-6} G$</th>
<th>$1 \times 10^{-5} G$</th>
<th>$5 \times 10^{-5} G$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha_1$</td>
<td>0.226</td>
<td>$2.8 \times 10^{12}$</td>
<td>125 $b^3$</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$\alpha_2$</td>
<td>0.150</td>
<td>$3.0 \times 10^{11}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$\alpha_3$</td>
<td>0.087</td>
<td>$6.7 \times 10^9$</td>
<td>$v = \text{const.} = 4 b^3$</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$\alpha_4$</td>
<td>0.075</td>
<td>$7.4 \times 10^{14}$</td>
<td>$v = \text{const.} = 1 b^3$</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

γ-peak undisturbed by interstitial atoms. A pronounced peak is present in the as deformed specimen state (fig.2). Figure 5 shows the γ-peak compared to a single Debye peak with activation parameters $H = 1.09 \text{ eV}$, $f_0 = 3 \times 10^{12} \text{ 1/s}$ (crystal orientation: [111]), after subtraction of an exponential damping background. The peak shape fits to a relaxation which is caused by a spectrum of relaxation times distributed according to a Gaussian function (see /10/), although the high temperature peak side is somewhat lowered against the Debye peak due to the annealing of the peak during warmup. The calculated Gaussian parameters are temperature dependent and amount to $1.0 - 1.6$.

The amplitude dependence of the spectrum above room temperature has been measured in detail, its evaluation and interpretation is still in progress.

4. Discussion.- In the following we shall discuss the observed relaxation processes in terms of the Kink Pair Formation (KPF) and the Diffusion of geometrical Kinks (KD) including possible interactions with point defects. In the stable specimen state 2 (fig.2, curve 2) intrinsic point defects are annealed out and unique results are obtained for 3 of the 4 subpeaks independent of the considered sample. The $\alpha_1$ process exhibits a strongly stress dependent activation volume. The numerical values are small compared to $b^2L$ ($L$ is the free dislocation length) but considerably larger than one atomic volume (table 1). These features are only accounted for by the KPF model /11/. Furthermore, in the KPF model a broadening of the damping peak with respect to a Debye peak is expected due to a distribution of dislocation lengths and internal stresses. At high external stresses $\sigma_a$ the Paré condition /12/ will be met by a growing number of dislocation segments of certain lengths, enhancing the peak and reducing its width. This is observed for $\alpha_1$ as explained in the previous section (fig.4). The subpeak $\alpha_1$ is increased by high temperature deformation, the specimen state in which tangles of
preferably 71°-dislocations are dominant in the dislocation structure /13/. We therefore attribute the subpeak \( \alpha_1 \) to the KPF in 71°-dislocations. The subpeaks \( \alpha_3 \) and \( \alpha_4 \) are enhanced by low temperature deformation. They exhibit similar activation energies and stress independent activation volumes which amount to the order of one atomic volume. This is expected for a dislocation motion via KD /1/. Due to the special symmetry of screw dislocations in the b.c.c. lattice the existence of different types of kinks in screw dislocations is conceivable /1/ even though as yet unconfirmed by experimental results. We attribute the subpeaks \( \alpha_3 \) and \( \alpha_4 \) to the diffusion of two different types of kinks in screw dislocations, most likely the kinks \( K_1^+ \) and \( K_1^- \) (in the notation of ref.1) with lowest formation energy. A distribution of diffusion lengths leads to overlapping distributions of the relaxation times for both processes and may account for the unequal preexponential factors \( f_0 \) stated in table 1 (this will be further outlined in /5/). The \( \gamma \)-peak is a symmetrical relaxation peak with a temperature dependent broadening relative to a single Debye peak. This is a necessary feature if the KPF mechanism is adequate. If one attributes the \( \gamma \)-peak to the KPF in screw dislocations all dislocation segments become mobile at the \( \gamma \)-peak temperature allowing for an irreversible rearrangement of the dislocation network. This may cause the disappearance of the \( \gamma \)-peak after annealing above the peak temperature which at least in the present case cannot be explained as pinning by interstitial atoms. The \( \beta \)-peak in our opinion is due to an interaction of deformation induced point defects with dislocation segments intrinsically relaxing in the temperature range of the \( \alpha \)-relaxation. By the disappearance of those point defects the dislocations are freed and the relaxation strength transferes into the \( \alpha \)-peak. Such an interaction peak may also be situated above the \( \gamma \)-peak (fig.2) as previously reported for iron /14/.

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