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DISLOCATION CORE DIFFUSION AND UNPINNING IN \( \alpha \)-Zr

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Abstract.- The high temperature, low frequency, internal friction spectrum of deformed and annealed \( \alpha \)-Zr is dominated at low strain amplitudes by a peak (\( P_2 \)) at \( \approx 600^\circ \text{C} \) (4 Hz). At higher strain amplitudes, two new peaks \( P_0 \) (\( \approx 480^\circ \text{C} \)) and \( P_1 \) (\( \approx 530^\circ \text{C} \)) appear. It is shown that \( P_0 \) is due to the thermally assisted unpinning of dislocations from oxygen interstitials while \( P_1 \) and \( P_2 \) are due to the longitudinal and transverse movements of the same defects in the dislocation core. \( P_2 \), previously attributed to grain-boundary relaxation, is a Snoek-Köster peak. A behavioural map of the temperature-strain amplitude plane can be constructed to facilitate the interpretation of amplitude-dependent results. It is shown that unpinning can be distinguished from longitudinal core diffusion, even when both are observed at the same temperature, provided the modulus defect is measured together with the internal friction.

1. Introduction.- The high temperature internal friction (\( \Delta \)) spectrum of \( \alpha \)-Zr is dominated by a peak (\( P_2 \)) at \( \approx 550^\circ \text{C} \) (1 Hz) (1-4). A plot of \( \ln f \) (\( f \) is frequency) vs. reciprocal peak temperature (1/\( T(P_2) \)) yields the relaxation enthalpy, \( \Delta H_R = 2.5 \text{ eV} \), and limiting relaxation time, \( \tau_0 = 10^{-16} \text{ s} \); \( P_2 \) is about twice as broad as a simple Debye peak (4). The height of \( P_2 \) (and \( \Delta \) throughout the high-temperature spectrum) is reduced by heat treatments which increase the grain size. Hence, it is not surprising that some authors (1,2) have attributed \( P_2 \) to grain-boundary relaxation. We have shown (4) that \( P_2 \) cannot be associated with ordinary grain boundaries as is the case with similar results on other pure metals (5). This paper outlines a detailed study of amplitude (\( \varepsilon \)) dependence in the vicinity of \( P_2 \). The results are attributed to thermally activated unpinning of dislocations, longitudinal core diffusion and transverse core diffusion. In the following, these phenomena are simply referred to as unpinning, LCD and TCD, respectively.

2. Material.- Measurements of \( \Delta \) in flexure (\( f = 4 \text{ Hz} \)) by both the free decay and constant-\( c \) techniques (6) were carried out on cold-rolled strip of nominally 5N pure Zr. Interstitial impurity concentrations (\( \mu \text{g/g} \)) were found by analysis to be: \( H < 5 \); \( N = 6 \); and 0, 17 to 23. Results similar to those presented below were obtained with specimens annealed for a few hours in the temperature range 500 to 700°C or vibration-annealed overnight (16 h) at 400°C in the pendulum (4).
3. Results. — $\Delta$ vs. $T$, $\varepsilon$ constant (figure 1)

(i) $\varepsilon \leq 2 \times 10^{-6}$ only $P_2$ is observed

(ii) $\varepsilon > 2 \times 10^{-6}$ $P_2$ is accompanied by $P_0$ ($= 480^\circ$C) and $P_1$ ($= 530^\circ$C) as shown by the difference curve in figure 1.

With increasing $\varepsilon$, both $P_0$ and $P_1$ shift to lower $T$ (as expected for thermally activated processes) while $P_2$ shifts slightly to higher $T$ (anomalous behaviour).

$\Delta$ vs. $T$, $\varepsilon$ constant (figure 2)

Fig. 1 Internal friction vs. temperature

Fig. 2 Internal friction vs. strain amplitude at selected constant temperatures.

(a) Region 1 (400 - 493°C). A peak develops and moves to lower $\varepsilon$ as $T$ increases. These curves are similar to those predicted by the Blair, Hutchison and Rogers (BHR) theory (7) based on the Teutonico, Granato and Lücke model (8) of dislocation unpinning. An important prediction of the BHR theory is that the modulus defect ($\eta$) as a function of $\varepsilon$ is $0.5 \Delta_0$, i.e. half the peak height, at the peak position, and saturates at $\eta = \Delta_0$ for large $\varepsilon$. This is confirmed experimentally in region 1 ($480^\circ$C) as shown in figure 3.

Fig. 3 Internal friction and modulus defect as a function of strain amplitude at 480°C in region 1 (left) and at 512°C in region 2 (right).
(b) Region 2 (493 - 580°C). A peak moves slowly to lower ε with increasing T. Its height after subtraction of the low ε background remains approximately constant. Although similar in shape to the peak in region 1, η/Δ₀ in region 2 is significantly greater than 0.5 as shown in figure 3. A plot of T vs. ε at the peak is a straight line. Its intercept at zero ε implies a relaxation enthalpy for P₁ of 1.86 eV. At 493°C the curve is made up of two peaks because of overlapping region 1 and region 2 behaviour.

(c) Region 3 (590 - 650°C). A peak moves back, anomalously, to higher ε with increasing T.

The complex behaviour in regions 2(P₁) and 3(P₂) is similar to the behaviour predicted theoretically for LCD and TCD. Lücke and Schlipf(9) were the first to show that both LCD and TCD give rise to relaxation peaks. Subsequently, their model for an ensemble of double-loops was thoroughly investigated by Winkler-Gniewek(10).

4. Comparison of Theory and Experiment. - Unpinning. Comparison of the BHR theory for very long segments with the curves of region 1 (P₀) yields estimates of the binding energy (U₀) and loop-length (κ): U₀ = 1.47 eV and κ = 2000 b (b is the Burgers vector). There is evidence that U₀ for impurity interstitials in the h.c.p. transition metals can be as high as μb³/6(11) (μ is the shear modulus). For α-Zr, μb³/6 = 1.55 eV, so that P₀ is consistent with unpinning from an impurity interstitial. Core Diffusion. LCD is similar to unpinning. A critical stress (σₙ) plays a role similar to the unpinning stress (σᵤ). Once σₙ is exceeded, there is an increase in the free dislocation-length. Thus, it is not surprising that the shapes of the curves for Δ vs. ε are similar in region 1 and region 2. LCD is governed by a relaxation time (τₗ) given by (10)

\[ \tauₗ = \bar{L}²/[Dₓ(n² + a²)] ; \bar{a}² = \sigma²b²\bar{L}³/(4TₖkT) \]  

\[ \bar{L} \] is the mean loop-length, Dₓ is the LCD coefficient, σ is the applied stress (σ = REε in flexure experiments; E is Young's modulus and R is the orientation factor), Tₖ = 0.5 μb² is the line tension, and k is Boltzmann's constant. The expressions for Δ and η are complex(10) but easily evaluated numerically. We have calculated Δ and η for homogeneous strain and for the strain distribution in our specimens(6). The calculated results agree qualitatively with the experimental results for region 2(P₁).

TCD gives rise to a superposition of elementary relaxation processes(10) in qualitative agreement with P₂. The effective relaxation time (τₜ) is given by

\[ \tauₜ = \bar{k}T(1-q²/\bar{L}²)/(2DₓTₖ) \]  

where q is the defect distance from the double-loop centre and Dₓ is the TCD coefficient. If, up to the given amplitude, the longitudinal distribution is 'frozen-in' (q fixed), then the peak is ε-independent up to that amplitude. However, if at a higher ε, longitudinal relaxation becomes possible, the TCD peak will show weak ε-dependence through (1-q²/\bar{L}²) in qualitative agreement with P₂.

We assume that the same defects cause P₀ and the P₁/P₂ complex. Then, identify-
ing $P_2$ ($H_R = 2.5$ eV, $\tau_0 = 10^{-16}$ s) with TCD and taking $\bar{F} = 2000$ b, $D_T = 210 \exp (-2.5 \text{ eV/kT})$. Similarly, identifying $P_1$ ($H_R = 1.86$ eV) with LCD, we find $D_L = 1570 \exp (-1.86 \text{ eV/kT})$. Thus LCD is much faster than TCD as expected intuitively. Also, the predicted variation of $T(P_2)$ with $\epsilon$ when there is longitudinal mobility (10), can show an increase with $\epsilon$ in the unexpected sense observed in region 3.

A comparison of region 2($P_1$) results and theory is shown in figure 4. The experimental values were multiplied by constant factors to force a fit at the peak position. This is equivalent to fixing $\Lambda \bar{F}^2$ ($\Lambda$ is the dislocation density) and $R$. At the highest $\epsilon$ values, the adapted theory is in good agreement with the results for both $\Lambda$ and $n$. At the lowest $\epsilon$ values the fit is poor. This is caused by comparing the oversimplified double-loop theory with results for multi-pin configurations. In such a case, $\sigma_c$ is very sharply defined, accounting for the almost step-like increase in $\Lambda$ observed experimentally.

The $T$ variation of the $\Lambda$ vs. $\epsilon$ peaks in region 2($P_1$) is shown in figure 5. Limiting values of $R = 1$ and $R = 2/(3\pi)$ are assumed and the results compared with those calculated. Considering the uncertainties in the parameters and the limitations of the theoretical model, figure 5 shows excellent agreement between theory and experiment.

The experimental results in figure 3 suggest that $n/\Lambda_0$ can be used to distinguish between the similarly shaped peaks due to unpinning and LCD. A comparison of
calculated ratios for $n/A_0$ with those found experimentally as tabulated below shows good agreement.

<table>
<thead>
<tr>
<th>Temp. °C</th>
<th>$n/A_0$ CALC.</th>
<th>$n/A_0$ EXPT.</th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td>480</td>
<td>0.50</td>
<td>0.50</td>
<td>Unpinning</td>
</tr>
<tr>
<td>512</td>
<td>0.58</td>
<td>0.59</td>
<td></td>
</tr>
<tr>
<td>531</td>
<td>0.60</td>
<td>0.57</td>
<td>LCD</td>
</tr>
<tr>
<td>550</td>
<td>0.70</td>
<td>0.67</td>
<td></td>
</tr>
</tbody>
</table>

5. **Map of the T-$\varepsilon$ Plane.** - In the BHR theory, the unpinning boundaries in the T-$\varepsilon$ plane are easily calculated (7). Also, the boundaries of 'instantaneous' core diffusion, $2\omega L = 1$ and $2\omega T = 1$ can be calculated. These boundaries and the experimental results are shown in figure 6. The arrow indicates the range of $\varepsilon$ covered experimentally. With the aid of this map, all of the complex and apparently anomalous results in figures 1 and 2 are easily interpreted.

![Fig. 6 Behavioural map of the T-$\varepsilon$ plane.](image)

If the root-mean-square vibration amplitude exceeds $\sigma_c$, defect redistribution will proceed, with a time constant $T_L$, at temperatures much lower than $T(P_1)$. This suggests an experiment to confirm the interpretations of $P_0$ and $P_1$ given above. It is a demonstration that either unpinning or LCD can be observed at the same temperature, depending upon the observation time. Another specimen was used and the results are given in figure 7. The full curves ($n/A_0 = 0.49$) traced in < 10 s are due to un-

![Fig. 7 Demonstration of unpinning (full curves) and longitudinal core diffusion (broken curves) at the same temperature.](image)
pinning. The broken curves \( \eta/\Delta_0 = 0.77 \) obtained by the constant-\( \epsilon \) method (90 s vibration at each step increase in \( \epsilon \)) are due to LCD. These results also demonstrate that the defects involved in \( P_0 \) and \( P_1 \) are indeed the same.

6. Discussion.— If \( D_B \) is the bulk-diffusion coefficient for the species of defect involved, it is expected, intuitively, that \( D_L > D_T = D_B \). Since \( D_T \) is very close to the diffusion coefficient for oxygen in \( \alpha\)-Zr(12) over the \( T \) range of \( P_0 \), \( P_1 \) and \( P_2 \), we identify the defects involved as oxygen interstitials. Lücke and Schlipf (9) have argued that \( D_L \) and \( D_T \) should be comparable and that the TCD boundary in figure 6 should be at lower temperatures than the LCD boundary. This is inconsistent with our results. The TCD peak \( (P_2) \) can be subsumed into the class of peaks attributed to the Snoek-Köster (13-14) relaxation. Our treatment of \( P \) is consistent with recent models of the Snoek-Köster relaxation, provided that the enthalpy of double-kink formation is negligible compared with the enthalpy of TCD.

Finally, it should be noted that three peaks similar to \( P_0 \), \( P_1 \) and \( P_2 \) have been observed in silver (15). Core redistribution of impurity interstitials appears to be a general phenomenon which contributes peaks to the low frequency, internal friction spectra of deformed metals.

REFERENCES