ULTRASONIC INVESTIGATION OF THE DISLOCATION STRUCTURE IN PLASTICALLY DEFORMED COPPER

H. Schmidt, D. Lenz, E. Drescher, K. Lücke

To cite this version:

HAL Id: jpa-00221092
https://hal.archives-ouvertes.fr/jpa-00221092
Submitted on 1 Jan 1981

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
ULTRASONIC INVESTIGATION OF THE DISLOCATION STRUCTURE IN PLASTICALLY DEFORMED COPPER

H. Schmidt, D. Lenz, E. Drescher* and K. Lücke

Institut für Allgemeine Metallkunde und Metallphysik RWTH Aachen, F.R.G.
*Institute of Fundamental Technological Research, Academy of Science, Warsaw, Poland

Abstract.- We have simultaneously measured the dislocation contributions to ultrasonic attenuation $\alpha_D$ and reduction of sound velocity $\Delta v/v$ between 10 and 200 MHz in deformed Cu. The samples were $\gamma$-irradiated in order to reduce the attenuation and extend the experimentally usable frequency range to higher frequencies. The results have been evaluated in terms of dislocation resonance damping. Quantitative data for the dislocation density and free dislocation loop length as function of $\varepsilon$ have been derived both for dislocations in tangles and in the cell interior.

1. Introduction.- Dislocation resonance (DR) is a frequently studied and theoretically well described internal friction phenomenon /1/. The predicted frequency ($f$)-characteristics of overdamped DR has been observed by MHz-attenuation $\alpha$ measurements /2/ and Hz- and kHz-experiments are expected to show the low $f$ DR behaviour for the modulus defect $MD = AL^2$ ($A$ = dislocation density, $L$ = dislocation loop length) and for the damping decrement $\delta \sim \alpha/f \sim B\!A^4L^4$ ($B$ = damping force constant). There is considerable experimental /3,4/ and theoretical /5,6/ support for the Granato Lücke DR-theory /7/ based on Koehler's vibrating string model /8/.

However, several problems with respect to the interpretation of $\delta$ and MD data have accumulated /9/. One problem is that $\delta \sim f$ as predicted by the GL-theory is often not observed in low $f$ experiments. Also in MHz experiments a puzzling deviation from the predicted $f$-dependence has been observed at low $f$, but only in pre-deformed samples. To our opinion an explanation of this effect may contribute to the clarification of other low $f$ problems.

We investigated the influence of deformation on $\delta$ and MD followed by $\gamma$-irradiation induced dislocation pinning. MHz pulse-echo experiments offer the unique chance to study the most revealing $f$-dependence of DR over a broad $f$-range (typically 5 to 200 MHz) in one and the same well defined sample volume in different pinning states.

2. Experimental.- A set of identical high purity (RRR $\approx$ 1000) Cu single crystals was deformed at room temperature by compression ($0.19% \leq \varepsilon \leq 27%$) along <111>. Sample preparation, $\gamma$-irradiation and measuring techniques for attenuation $\alpha$ and sound velocity $v$ are described in /10/.

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jphyscol:1981550
3. Results.- Fig. 1a shows $\alpha(\varepsilon)$ measured at 10 MHz. Curve D represents the $\alpha$ values 30 min after deformation (time for control of sample quality and for transducer bonding): $\alpha$ increases with $\varepsilon$ to a maximum at $\varepsilon = 0.4\%$, decreases then and becomes nearly constant at $\varepsilon > 5\%$. An (1h, 373K)-anneal reduces $\alpha$ considerably (curve R). The $\gamma$-irradiation ($T_{\text{IRR}} = 320\,\text{K}$, followed by an (1h, 353K)-anneal) further reduces $\alpha$ towards the minimum value $\alpha_B$ ("non dislocation background") which could not be reduced further by additional irradiation. For $\varepsilon < 1\%$ the $\alpha_B$ values are the same as for background irradiated standard samples /11/. The $\alpha_B$ increase at $\varepsilon > 1\%$ is due to sound wave scattering by deformation induced inhomogeneities (kink bands, as shown by metallographic analysis) and is not due to dislocations (as proved by additional irradiation). Fig. 1a demonstrates that the $\alpha$-maximum around $\varepsilon = 0.4\%$ is caused by dislocations which become pinned with increasing $\gamma$-dose ($\varnothing_\gamma$). For comparison fig. 1b shows the sound velocity $v$ 30 min after deformation (D) and after the "background" dose (curve $v_0$). The $v(\varepsilon)$ data after intermediate $\varnothing_\gamma$ (omitted for sake of clarity) approach very (R: 1h, 373K) and with increasing $\varnothing_\gamma$ the $v_0$ curve (which as $\alpha_B$ cannot be changed by further irradiation). At $\varepsilon < 1\%$ $v_0$ is given by the longitudinal sound velocity $v_{L<111>}$ of the ideal ("dislocation free") Cu crystal /10/. The $v_0$-decrease at $\varepsilon > 1\%$ is due to increasing deformation induced misorientation ("crystallographic modulus defect") as shown by x-ray measurements. Since $v_{L<111>}$ represents the maximum fcc sound velocity any deviation from $<111>$ results in a velocity reduction.

4. General Discussion.- We consider the evaluation of $\alpha(f)$ and $v(f)$ (fig.1a,b shows only the 10 MHz results) to be the key to further understanding. Fig.2 shows the complete data set for the decrement $\delta$ (open symbols) and the modulus defect MD (solid symbols)

$$\delta \text{ [Neper]} = 0.115 \cdot \left\{ \alpha(f) - \alpha_B(f) \right\} \text{[dB/\mu s]/f[MHz]}$$

$$\text{MD} \equiv \Delta M/M_0 = \left( M_0 - M(f) \right) / M_0 = 2 \left( v_0 - v(f) \right) / v_0$$

for the 1% deformed sample (D), after recovery (R) and irradiation ($\varnothing_\gamma$). Since for standard crystals the pinning behaviour is quantitatively described by the GL-theory /10/ we compare the present data on deformed samples also with the theoretical GL- ($\delta, \text{MD}$)-profile (GLP). Each GLP consists of a $\delta$-branch (curve showing the over-
damped resonance maximum, arrows in fig.2) and a MD-branch which are interlinked (large open circles in fig.2) because of the Kramers-Kronig relation/12/. We use the GLP for the exponential loop length distribution which is invariant against random pinning/10/. As fig.2 shows the GLP fits our high $\Phi_1$ data very well. However, for the $\Phi_1=800\mu$Ah data and especially for the state R and D data an increasing discrepancy exists for the decrement at low f: The measured $\delta$ values considerably exceed the theoretical curves (e.g. by the factor 1.6 at 10 MHz in state D) whereas the MD data are much better described by the respective GLP. According to the GL-theory any change of the mean loop length $L$ due to pinning causes the GLP to move downwards a slope-l line. Therefore the present GLP fits are strongly supported by the observed slope-1 shift (dashed dotted line through $\delta_{\text{MAX}}$ (arrows) in fig.2) between $\Phi_1=800$ and 15000 $\mu$Ah. Accordingly, we fit our R and D data (for state D only 10 MHz data are available because of extremely high $\alpha$ (c.f. fig.1a)) with the GLP shifted upwards the same slope-1 line. The observed low f discrepancy cannot be removed by a modification of the exponential $L$-distribution function (since this would deteriorate the good ($\delta$,MD) fits at high $\Phi_1$ and also the fair MD fit at low f in state R and D. The discrepancy is observed after RT deformations between 0.1 and 10%; it is still observed after standard annealing at 923K (fig.3;c.f. also fig.3 in /10/). However, as fig.3 shows the discrepancy is not observed in as grown crystals. Thus we conclude that the discrepancy is caused by deformation.

It has been shown by TEM that polyslip oriented (e.g. $<111>$) crystals already at low $\epsilon$ develop a dislocation structure consisting of high $\Lambda$ areas (tangles) and low $\Lambda$ areas (cell interior)/13,14/. Consequent-

![FIG 2 Frequency dependence of dislocation modulus defect (solid symbols) and decrement (open symbols) after deformation (D), recovery (R) and $\gamma$-irradiations ($\Phi_1$) fitted by theoretical KGL-curves.](image1)

![FIG 3 $\delta(f)$ before ($\bullet$) and after standard treatment ($\circ$) compared with KGL-curves. Note the discrepancy at 10 MHz in the deformed sample.](image2)
ly we base the further evaluation on such a structure: In addition to the \((\delta_1, MD_1)\) component (which is described by the solid GLP in fig.2) responsible for most of the observed \(\delta\) and MD a second dislocation resonance component \((\delta_2, MD_2)\) with \(\delta_2 = \delta - \delta_1\) (e.g. dashed GLP for state D with \(f_{\text{MAX}_2} \approx 1 \text{ MHz}\)) accounts for the \(\delta\)-discrepancy at low \(f\). As seen by the dashed GLP in fig.2 the accompanying \(MD_2\) is hardly observable \((MD_2 << MD_1)\). Furthermore fig.2 shows that \((\delta_2, MD_2)\) rapidly decrease during recovery and irradiation (800 \(\mu\text{A}\)), i.e. the component 2 becomes rapidly reduced by pinning in comparison with component 1. Since \(f_{\text{MAX}} \sim \Lambda^2\) and \(f_{\text{MAX}} \sim 1/L^2\) \cite{7} it follows from fig.2 that \(L_2 \approx 10L_1\) and \(\Lambda_2 \approx 0.01 \Lambda_1\). Consequently we attribute the component 2 (caused by long dislocations of low \(\Lambda\)) to the cell interior dislocations and component 1 (caused by short dislocations of high \(\Lambda\)) to the dislocations in the tangles. The rapid pinning of component 2 is a consequence of the small \(\Lambda\) and long \(L\) of the cell interior dislocations competing for the deformation- and irradiation induced point defects (the dislocation/point defect drainage volume \(V_D\) per loop length \(L\) is given by \(V_D \sim L/\Lambda\) i.e. \(V_D/\sqrt{2} = 10^3\) in state D). The resistance of the observed discrepancy against heat treatment indicates that the tangled structure is not removed (e.g. by 4\(h\), 923\(^\circ\)C annealing). This is in agreement with TEM results which show that for \(\varepsilon < 0.5\%\) the deformation induced dislocation structure is not significantly changed by our standard anneal /15/.

5. Quantitative evaluation and discussion.- The described \((\delta, MD)\) fit procedure was done for the samples with \(\varepsilon \leq 6.2\%\) \cite{16} (for \(\varepsilon > 6.2\%\) the measured \(\delta\) and MD values are too small for reliable quantitative evaluation (c.f. fig.1a,b)). From the GL-theory \(\Lambda\) and \(L\) are calculated according to

\[
\Lambda = 15.66 \frac{\delta_{\text{MAX}} f_{\text{MAX}} B \Omega G b^2}{\Lambda_1}
\]

\[
L = \sqrt{0.113} \frac{C f_{\text{MAX}} B}{\Lambda_2}
\]

with \(\delta_{\text{MAX}}, f_{\text{MAX}}\) coordinates of the \(\delta\)-maximum, \(B = 6.5 \times 10^{-5} \text{Ns/m}^2\), \(\Omega = 0.088\) orientation factor, \(G = 4.08 \times 10^{10} \text{N/m}^2\) shear modulus, \(b = 2.55 \times 10^{-10} \text{m}\) Burgers vector and \(C = 5.5 \times 10^{-10}\) line tension (values from /2/).

Dislocation density.- With equ.(3) we obtain \(\Lambda_1 [\text{cm}^{-2}]= 1.5 \times 10^9 \varepsilon [\%]\) (fig.4). This result (being in quantitative agreement with TEM measurements /13,14/ proves that component 1 is caused by the tangled dislocations. In contrast to \(\Lambda_1\) the cell interior density \(\Lambda_2\) is independent of \(\varepsilon\) for small \(\varepsilon\) and close to the dislocation density of our undeformed crystals.

Dislocation density in tangles \((\Lambda_1)\) and in the cell interior \((\Lambda_2)\) as function of deformation.
\( \Lambda_0 = 6 \times 10^6 \text{cm}^{-2} \). These \( \Lambda_2 \) results add information to TEM work which is mostly confined to high dislocation density areas.

**Dislocation loop length.** Since \( L \) is determined by different pinning effects we discuss the pinning in terms of \( n \) the number of pinning points (PP) per cm dislocation \( n = 1/L = 2n \). In a well annealed crystal we expect \( n = \Delta n + n + n \Lambda \) with \( n \Lambda = \xi \Lambda \) the network PP (network length \( L_N \) shortened by mutual dislocation cutting); \( n \) the statistical impurity PP in the lattice; \( \Delta n \) the enrichment of impurity PP at dislocations (due to e.g. Cottrell interaction). For the deformed crystals we take \( \Delta n = 0 \) (it seems unlikely that in \( 1 \text{h at 373K} \) Cottrell clouds are formed around fresh dislocations), and additionally account for the deformation produced PP \( n_e \) and the irradiation induced PP \( n_y \):

\[
n = n_c + n + n_e + n_y
\]  

We assume (i) \( n_c \) to be independent of \( \varepsilon \) and given by \( n_c = 1/L_c = 2.5 \times 10^4 \text{cm}^{-1/17} \); (ii) \( n \Lambda = \xi \Lambda \) (network model); (iii) \( n_e = k N(\varepsilon)/\Lambda \); with \( N(\varepsilon) = n_L \Lambda^2 \) the lattice concentration of deformation induced point defects \( /18/ \) we get \( n_e = k n_L \Lambda = n \Lambda \); (iv) \( n_y = o \delta /\Lambda \) with \( \delta \) the effective cross-section for irradiation PP. The \( 1/\Lambda \) factor in (iii) and (iv) accounts for the drainage competition (which reduces \( n_e \) and \( n_y \) with increasing \( \Lambda \) in the case of complete drainage). Then for the deformed and irradiated crystals we obtain

\[
n = n_c + \xi \Lambda + n \Lambda + \delta /\Lambda
\]  

Fig. 5 shows a plot of \( \text{(n-n}_c)/\sqrt{\Lambda} \) vs \( \sqrt{\Lambda} \) for component 1 (tangled dislocations) as suggested by equ.(6) for \( \theta = 0 \). We postpone the discussion of the irradiation pinning \( /19/ \). The obtained linear fit (yielding \( \xi = 0.75, \eta = 4 \times 10^{-5} \text{cm} \)) shows that mutual dislocation cutting as well as pinning by deformation induced point defects contribute to \( L(\varepsilon) \). The value for \( \xi \) is in good agreement with \( \xi = 0.6 \) for the simple cubic network \( (\Lambda^2 = 3) \); from the \( n \) value it follows that \( k = 10^{-3} \) i.e. only \( 0.01\% \) of the deformation induced defects are converted into PP \( /19/ \). At \( \varepsilon > 0.3\% \) the \( L(\varepsilon) \)-shortening overcomes the influence of \( n_L(c) \) on \( \delta \) and MD (fig.1a,b).

For a single component the \( \delta(\Lambda), \text{MD}(\Lambda) \) behaviour can be given for the low \( f \) range \( (f < f_{\text{MAX}}) \) with \( L = 1/n \) and equ.(6, \( \theta = 0 \)) we obtain

\[
\delta_{\text{LOW}} \sim \Lambda^4 = (n_c + \xi \Lambda + n \Lambda)^4
\]

\[
\text{MD}_{\text{LOW}} \sim \Lambda^2 = (n_c + \xi \Lambda + n \Lambda)^2
\]

At small \( \Lambda \) both \( \delta \) and MD increase \( \sim \Lambda \), then

FIG 5

Fit for evaluation of \( \xi \) and \( \eta \) (L-shortening coefficients) in equ.(6).
pass through a maximum (given e.g. for the MD by $\hat{\lambda}_{MD} = n_C/\eta$) and decrease at high $\Lambda$ as $\rho_1^{LIM}/LIM = 1/\Lambda^3$, $\rho_2^{LIM}/LIM = 1/\Lambda$. We expect that equs. (7) and (8) hold for Hz- and kHz experiments. In these cases (c.f. fig.2) the measured DR damping is almost completely caused by the cell interior dislocations i.e. $\delta = \delta_2$; however, an excess modulus defect due to the tangled dislocations should be observed $MD = MD_2 + MD_1$. We point out that for $f > f_{MAX}$ (and if different DR components contribute to $\delta$ and $MD$ as in the present case) the complete GLP (including separation into components) has to be used for the evaluation. We note that Schlipf /20/ considering dislocation interaction effects (mutual cutting and point defect generation) arrives from different arguments at the same $\Lambda$-dependence as given in our equ.(6), $\Theta_0=0$. A more detailed discussion of the present $\xi$ and $\eta$ values and of irradiation pinning in deformed Cu will be published elsewhere /19/.

Work supported by the DFG (SFB 125).

References

/5/ A. Seeger, P. Schiller: Acta Met. 10, 348 (1962)
/7/ A.V. Granato, K. Lücke: J. Appl. Phys. 27, 583 (1956)
/10/ D. Lenz, K. Lücke. H. Schmidt: this volume
/13/ P. Ambrosi, E. Göttler, Ch. Schwink: Scripta Met. 8, 1093 (1974)
/15/ M.J. Le Héryc: Memoires Scientifiques Rev. Metallurg. 9, 769 (1967)
/19/ D. Lenz, H. Schmidt: to be published
/20/ J. Schlipf, R. Schindlmayr: this volume