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EVALUATION OF DISLOCATION PINNING EFFECTS FROM SIMULTANEOUS MEASUREMENTS OF ATTENUATION AND SOUND VELOCITY IN COPPER

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Abstract.—The frequency dependence (10 to 200 MHz) of the sound velocity and of the ultrasonic attenuation \( \alpha \) has been measured on high purity Cu during 3 MeV \( \gamma \)-irradiation at room temperature. The results are evaluated with respect to dislocation effects which under the present conditions cause the major contributions to the frequency dependent reduction of sound velocity \( \Delta v_D/v_0 \) and attenuation \( \alpha_D \). The \( \gamma \)-dose dependence of \( \Delta v_D/v_0 \) and \( \alpha_D \) is in complete accordance with the Granato-Lücke theory of dislocation resonance damping. It is shown that simultaneous measurements of \( \Delta v_D/v_0 \) and \( \alpha_D \) strongly improve the accuracy of MHz-investigations of dislocation damping effects.

1. Introduction.—In high purity single crystalline fcc metals and in dilute alloy crystals (typically < 10\(^2\) ppm) the ultrasonic attenuation \( \alpha \) in the MHz f-range is mainly caused by anelastic dislocation effects attributed to overdamped dislocation resonance /1/. The dislocation decrement \( \delta \) is accompanied by the dislocation modulus defect \( MD \) which is measured as a reduction and dispersion of sound velocity. Anelasticity due to dislocation resonance has been successfully described on the basis of Koehlers vibrating string model /2/ by the Granato-Lücke theory /3/. This theory yields from \( \delta(f) \) - and \( MD(f) \) -data quantitative information about interesting dislocation properties (e.g. dislocation density, dislocation loop length, dislocation damping force factor).

Simultaneous measurements of damping and modulus are a standard technique in the Hz- and kHz-ranges whereas in most MHz investigations only the attenuation is measured. One reason is that the dislocation attenuation effects are large and easy to be measured as function of f by the pulse-echo technique whereas the related \( \Delta v \)-effects are small and not easily measured as function of frequency. By the present investigation we try to find out whether simultaneous \( \delta(f) \) - and \( MD(f) \) -measurements improve quantitative evaluation of dislocation damping phenomena.

2. Experimental.—The investigated sample was of high purity Cu (<5ppm unknown impurities, residual resistivity ratio RRR=2200), spark-erosion cut from a \(<111>\)-oriented Bridgeman crystal, carefully lapped (plan-
parallelism and flatness of endfaces < 10^{-4} cm/cm, crystallographic orientation difference < 0.25 degrees from \langle 111 \rangle). After this preparation the sample was pre-annealed (4h, 650°C, 1.3x10^{-3} Pa), deformed to increase the dislocation density (0.1% compression along \langle 111 \rangle) and finally annealed (4h, 650°C, 1.3x10^{-3} Pa) in order to reduce and stabilize the dislocation density and structure respectively /4/.

The ultrasonic phase velocity \( v \) was determined from \( v = n \cdot 2s/t \) with \( s \) = sample length in direction of sound propagation, \( t \) = the travel time and \( n \) the number of round trips of the sound pulse between the endfaces of the sample. The time \( t \) was measured at 25 ± 0.05°C between 10 and 200 MHz by the pulse-overlap technique /5/ (absolute accuracy 5x10^{-5} and a sensitivity of 1x10^{-6}). The attenuation was simultaneously measured by the standard pulse-echo technique /6/. Matec equipment was used. For generation and detection of the sound pulses a compressional quartz-transducer (X-cut, 10 MHz fundamental frequency) was bonded onto the sample by "Nonaq". The \( \gamma \)-irradiation was produced by a Van De Graaff electron accelerator in an Au-target (Frenkel defect production rate 5.5x10^{9} FD/cm^{3} sec /7/).

3. Results.- Fig. 1 shows the frequency dependence of sound velocity \( v(f) \) before (open circles 0) and after (crossed circles \( \oplus \)) heavy \( \gamma \)-irradiation (\( \Phi_{\gamma} = 2500 \) \( \mu \)Ah). This \( \gamma \)-dose completely suppresses dislocation damping due to dislocation pinning by the irradiation induced point defects. Fig. 1 shows that this pinning increases \( v \) between 1.5 \% (10 MHz) and 0.3 \% (90 MHz). Because of too high initial attenuation no measurements were possible above 90 MHz in the unirradiated state. Before irradiation the velocity increases with increasing frequency in the sense of "normal" dispersion. However, after irradiation the velocity decreases from 10 to 70 MHz and becomes \( f \)-independent at \( f > 70 \) MHz. This "anomalous" dispersion behaviour is due to phase effects caused by sound diffraction in the soundfield of the transducer /8/. After correction for this effect and influences of the transducer-bond /9/ (- 1 \( \mu \)m Nonaq thickness) shown in fig.1 by the solid circles and crosses the velocity of the irradiated sample (crosses) is frequency independent within experimental accuracy (ideal elastic body). This velocity \( v_{o} \) is the reference velocity for
the separation of the wanted dislocation contribution \( \Delta v_D(f) \) from the measured velocity \( v(f) \) according to \( \Delta v_D(f) = v_0 - v(f) \).

Fig.2a shows the frequency dependence of attenuation \( \alpha(f) \) for different \( \gamma \)-doses \( \Phi_\gamma \) up to 2500 \( \mu \)Ah. Since this dose completely suppresses the dislocation damping \( \alpha_B \) is the background-attenuation of the "dislocation free" crystal /4/.

For the same \( \gamma \)-doses as in fig.2a fig.2b shows the \( v(f) \)-dependence (corrected for diffraction and bond effects). Comparing fig.2a and 2b it is seen that \( \gamma \)-irradiation (i.e. pinning) decreases the attenuation and increases the velocity towards the limiting values \( \alpha_B \) and \( v_0 \) resp.. In contrast to attenuation (showing ~90% dislocation effect) the effect on sound velocity is only in the 1 \( 0/00 \) range.

4. Discussion.- Fig.3 shows the frequency dependence of the dislocation decrement (open symbols)

\[
\delta[\text{Neper}] = 0.115 \cdot 
\cdot (\alpha(f) - \alpha_B(f)) [\text{dB}/\mu\text{sec}] / f [\text{MHz}] \tag{1}
\]

and dislocation modulus defect (solid symbols)

\[
MD = \Delta M / M_0 = \left[ M_0 - M(f) \right] / M_0 = 2 \left[ v_0 - v(f) \right] / v_0 \tag{2}
\]

Both quantities are compared with the corresponding theoretical \( \delta(f) \)- and \( MD(f) \)-curve respectively given by the GL theory for an exponential distribution of loop length /3/. With respect to the quality of the fit we point out that the two theoretical curves used to fit the set of measured data (\( \delta(f) \) and \( MD(f) \)) for a given state of the sample (\( \Phi_\gamma \)) are not independent. They must be thought as being linked together at one common point (large circles in fig.3) since dispersion and absorption in a dissipative medium are linked together by the Kramers-Kronig relation /10/. We mention furthermore that the irradiation effect is only due to dislocation pinning since the initial concentration of \( \gamma \)-pro-
duced defects is very low (< 5x10^-4 ppm) which excludes any measurable bulk effect on MD. Keeping both aspects in mind our irradiation experiment yields the strongest test of theory so far: our results (fig.3) are in excellent agreement with the GL-theory of dislocation resonance.

We now use the GL-theory for evaluation of dislocation pinning effects. The coordinates of \( \delta(f) \)-maxima (arrows in fig.3) are

\[
\delta_{\text{max}} = 0.565 \Omega G b^2 \Lambda L^2 / C \quad (3) \\
f_{\text{max}} = 0.113 C / L^2 B \quad (4)
\]

(valid for the exponential loop length distribution, \( L \) = mean free loop length, \( \Omega \) = orientation factor, \( G \) = shear modulus, \( b \) = Burgers vector, \( C \) = line tension, \( B \) = damping force factor /11/, \( \Lambda \) = dislocation density). The high frequency \( (f >> f_{\text{max}}) \) asymptote of the \( \delta(f) \)-curves (dashed-double-dotted line of slope -1 in fig.3) is given by

\[
\delta_{\text{HIGH}} = 0.405 \Omega G b^2 \Lambda / B f
\]

The corresponding MD\( (f) \)-curves can be characterized by the coordinates of the point \( MD^* = 0.5 \) \( MD_{\text{LOW}} \) (arrows on dashed curves in fig.3) where \( MD_{\text{LOW}} \) is the maximum modulus defect measured at \( f << f_{\text{MAX}} \)

\[
MD^* = 0.25 \Omega G b^2 \Lambda L^2 / C \\
f^* = 0.123 C / L^2 B \quad (6)
\]

We assume that irradiation pinning can be considered as adding pinning points to the dislocations in a random way. This does not change the initial exponential loop length distribution but only decreases its mean value \( L \). This \( L \)-change shifts the interlinked \( \delta(f) \)- and MD\( (f) \)-curves e.g. along the common line of slope -1 (dashed dotted line in fig.3) as can be directly seen from equus. (3), (4) and (6), (7) resp.

Since \( \delta_{\text{HIGH}} \) (equ. (5)) is independent of \( L \) the high frequency asymptote
is the same for all $\delta(f)$-curves irrespective of $L$. Fig.3 proves the good agreement between measured irradiation influence and theoretical prediction for statistical $L$-changes.

The number of additional irradiation induced pinning points per initial loop length $L_0$

$$p = \left( \frac{L_0}{L(\varphi_\gamma)} \right) - 1 \quad (8)$$

($L(\varphi_\gamma)$=mean loop length after irradiation with dose $\varphi$) can be calculated alternatively from the measured shifts of $\delta(f)$- or MD($f$)-curves according to

$$p = \sqrt{\frac{f_{\text{MAX}}(\varphi_\gamma) / f_{\text{MAX}}(0)}{f_{\text{MAX}}^*(\varphi_\gamma) / f_{\text{MAX}}^*(0)}} - 1 = \sqrt{\frac{f_{\text{MAX}}(\varphi_\gamma) / f_{\text{MAX}}^*(0)}{f_{\text{MAX}}^*(\varphi_\gamma) / f_{\text{MAX}}(0)}} - 1 \quad (9)$$

(c.f. equus.(4) and (7)). We point out that $p$ is obtained without resort to the quantities $C, B, \Lambda$ etc. in equus.(3), (4), (6), (7). As an example fig.4 shows the $\gamma$-dose dependence of $p$ derived from fig.3 and equ.(9).

The resulting $p(\varphi_\gamma)$ behaviour is in accordance with earlier results /7/ by showing the unexpected $p$-saturation at relatively low values ($\approx 1.5$).

As mentioned above from purely theoretical reasons the present technique of simultaneous $\delta$ and MD measurements (which more than doubles the experimental expenditure) yields no doubling of the information gained. However, we like to mention several advantageous applications of the present technique: i) at low frequencies ($f < f_{\text{MAX}}$) the modulus defect attains its maximum value $MD_{\text{LOW}}$ which can be measured easiest whereas the decrement decreases to very low values (since $\alpha = \alpha_B$). This situation is expected for impure samples (short $L$, $f < f_{\text{MAX}}^* - 1/L^2$, equ.(4)). The contrary situation is met with pure samples (large $L$, $f > f_{\text{MAX}}^*$) which exhibit relatively small MD at high $f$ as compared with $\delta$. In this case attenuation measurements are more suitable than MD-measurements. ii) An even more important advantage of MD-measurements occurs at low temperatures: In contrast to attenuation the sound velocity is not influenced by the phonon-electron interaction to any presently measurable degree /12/. Thus measurements of the dislocation modulus defect are imperative at temperatures where the attenuation is dominated by phonon-electron absorptions /13/ (e.g. with Cu, $RRR=1000$, 50 MHz at $T<80K$). iii) For a simple Debeye relaxation (or $\omega \tau << 1$ for an exponential distribution in $\gamma$ $\delta/MD = m \cdot f \cdot \tau$ ($m$=numerical constant, $\tau$=relaxation time /1,10/). In these cases measurements of $\delta$ and MD at a single fixed frequency $f$ directly
yield \( \tau \) as function of e.g. temperature, deformation, pinning... without knowledge of the relaxation strength of the process (however, such measurements are misleading if more than one anelastic process contributes to the measured \( \delta \) and MD resp.).

5. **Conclusions.** - 1. MHz-sound velocity measurements \( v(f) \) of the modulus defect caused by dislocations are valuable in addition to measurements of attenuation if they are done with an accuracy < \( 5 \times 10^{-5} \) over about one decade in frequency. The \( v \)-measurements are especially valuable at low frequencies and low temperatures where they provide information about dislocation effects not easy to obtain from attenuation. 2. The present investigation shows that the frequency dependence of dislocation decrement as well as the frequency dependence of dislocation modulus defect is in agreement with dislocation resonance theory. The observed loop length distribution is given by the exponential distribution function. The same distribution holds for changes of the mean loop length (produced by irradiation pinning). 3. Simultaneous measurements of dislocation attenuation and modulus defect provide a more stringent quantitative evaluation of dislocation pinning experiments (as compared with the less difficult attenuation measurement). This advantage is very important for quantitative measurements of dislocation parameters \( \Lambda \) and \( L \) as function of temperature and/or plastic deformation /14/.

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