

# Amplitude-dependent internal friction and dislocation inelastic strain in zinc and aluminium single crystals

S.N. Golyandin and S.B. Kustov

A.F. Ioffe Physico-Technical Institute, Russian Academy of Sciences, Politekhnicheskaya 26, 194021 St. Petersburg (Russian Federation)

## Abstract

An experimental study of the amplitude-dependent internal friction, Young modulus defect and reversible inelastic strain in Al and Zn single crystals has been performed. The investigations were carried out in the low frequency (0.005–1 Hz) range. The amplitude–frequency spectra of the internal friction and Young modulus defect were analysed taking into account the quasi-static athermal component due to dislocation–dislocation interaction or internal stress field hysteresis and the thermally activated component due to dislocation interaction with point defects. The marked amplitude hysteresis and time dependence of the internal friction in Zn single crystals were attributed to point defect redistribution in the glide planes of mobile dislocations. The analysis of stress–inelastic strain hysteretic loops for Zn single crystals confirms the Kressel–Brown model of quasi-static internal stress field hysteresis.

## 1. Introduction

In previous papers dealing with the amplitude–frequency spectra of the amplitude-dependent internal friction (ADIF), Young modulus defect (YMD) and reversible inelastic strain in alkali halides [1, 2], the conclusion was reached that at least two mechanisms linked to different levels of defect structure contributing to the amplitude-dependent inelastic behaviour of alkali halides exist. It was shown that the origin of the first component was internal stress (IS)  $\sigma_i$  hysteresis or dislocation ensemble interaction. This component was shown to be strain rate independent, *i.e.* athermal, and quasi-static, *i.e.* existing at the lowest investigated frequencies. The second component was caused by the strain-rate-dependent effective stress linked to dislocation–point defect (PD) interaction. It was shown that the dependence of the effective stress  $\sigma^*$  on the inelastic strain rate  $\dot{\epsilon}_{in}$  was  $\dot{\epsilon}_{in} = K(\sigma^*)^m$ , where  $K$  and  $m$  are similar to those obtained from the strain rate sensitivity of macroscopic deformation. On the basis of these results a phenomenological model was proposed and numerical calculations of different mechanism contributions were carried out over wide frequency and amplitude ranges [2].

In this work the conclusions of refs. 1 and 2 were checked at low frequencies for h.c.p. (Zn) and f.c.c. (Al) crystals. These crystals, with essentially different dislocation structures formed by plastic prestrain, were also chosen for experimental study of the mechanism

of dislocation–dislocation interaction contributing to the ADIF. A detailed comparison of ultra- and infrasonic data and the quantitative separation of ADIF components will be given in ref. 3.

## 2. Experimental details and results

The Al and Zn single crystals used were of 99.999% and 99.997% purity respectively. The samples were spark cut, etched to remove the surface layer and annealed either for 5 h at 773 K (Al) or for 4 h at 633 K (Zn). The Al single crystals had a “soft” orientation with the compression axis near the [035] direction. The Zn single crystals were oriented for single basal slip (0001){11 $\bar{2}$ 0} with a Schmid factor of 0.5. The experimental technique used direct registration of stress–strain hysteretic loops in the course of cyclic loading of samples in compression at room temperature. A capacitance strain gauge with a resolution of approximately  $10^{-8}$  was used for strain measurements. The strain amplitude range was  $5 \times 10^{-7}$ – $10^{-5}$  and the frequency range for the largest amplitudes was  $5 \times 10^{-3}$ –1 Hz. The samples had dimensions of approximately  $5 \times 5 \times 13$  mm<sup>3</sup> and were plastically prestrained during the tests. The experimental details can be found in ref. 1.

Figure 1 shows the amplitude and frequency dependences of the ADIF and YMD for an Al single crystal prestrained to 0.3%. Figure 2 shows the cor-

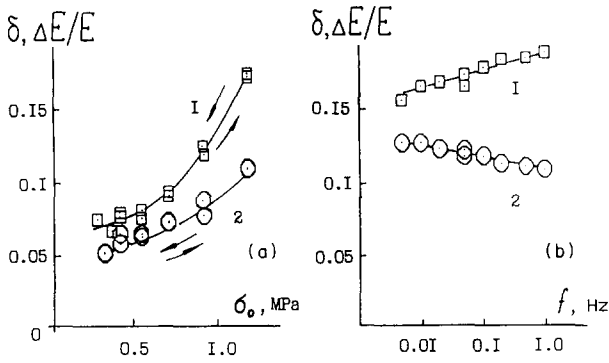


Fig. 1. Decrement (1) and YMD (2) for Al crystal vs. (a) stress amplitude at frequency 0.5 Hz and (b) frequency at stress amplitude 1.2 MPa.

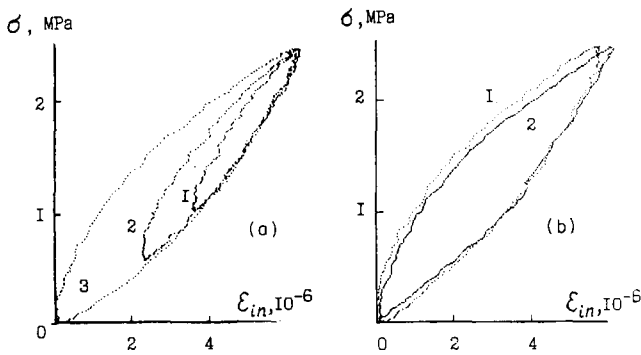


Fig. 2. Stress–inelastic strain HLs for Al crystal: a, frequency 0.5 Hz, stress amplitudes 0.7 (1), 0.9 (2) and 1.2 MPa (3); b, stress amplitude 1.2 MPa, frequencies 0.5 (1) and 0.005 Hz (2).

responding stress–inelastic strain hysteresis loops (HLs). The HLs for different stress amplitudes (Fig. 2a) are shifted to a single top. The dependences were measured for a statically loaded sample with a superimposed oscillatory sinusoidal tensile starting stress. One can note the following features.

(1) No hysteresis is observed in the ADIF and YMD for increasing and decreasing stress amplitudes (Fig. 1a).

(2) The symmetry of the HLs (Fig. 2a) is the same as for alkali halides and metals with different lattice structures (see ref. 1 and references cited therein) and is consistent with “unlocalized friction” models of dislocation hysteresis rather than with “breakaway” models.

(3) The unloading (and, because of the HL symmetry, also the loading) parts of the HLs measured at different stress amplitudes practically coincide. As for alkali halides [1, 2], the largest difference can be observed near the vertices of the loops (Fig. 2a).

(4) The frequency dependences are weak and show the decrement increasing and the YMD decreasing with increasing frequency. The behaviour of the ADIF and YMD is illustrated by the HLs measured at 0.005 and 0.5 Hz (Fig. 2b). It is clearly seen that the strain value is slightly increased at 0.005 Hz, while the area

of the HL is decreased as compared with the HL at 0.5 Hz.

A pronounced time dependence of the ADIF and YMD was observed in Zn samples. Figure 3 shows the amplitude dependences of the ADIF and YMD for a Zn sample prestrained to 0.08%, where every experimental point was obtained after 20 cycles of oscillations, i.e. under steady state conditions. Marked hysteresis in the ADIF and YMD for increasing and decreasing stress amplitudes is visible. However, measurements repeated after holding the sample at a constant stress for a few minutes reproduce in detail the data in Fig. 3. The most significant hysteresis is observed in the low amplitude range. The ADIF maximum occurs for decreasing stress at an amplitude of approximately 50 kPa.

Figure 4 shows the corresponding steady state HLs measured for increasing and decreasing stress amplitudes in the vicinity of the ADIF maximum. It is clearly seen from Fig. 4a that the HL for each subsequently increased amplitude accumulates more strain than the previous HL at the same stress value. This effect becomes less pronounced with increasing stress amplitude. For decreasing stress amplitude (Fig. 4b) the loading parts of HLs 1–4 lie closer to each other which than is typical for crystals where time effects and amplitude hysteresis

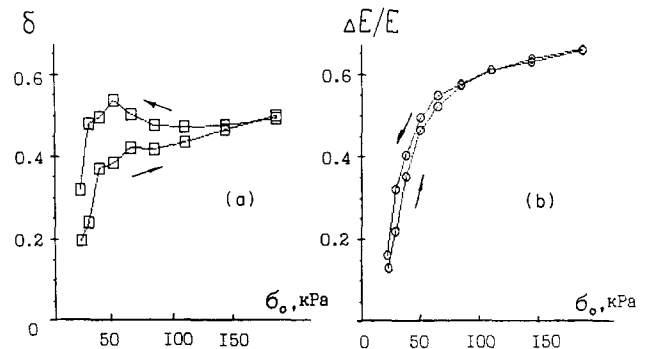


Fig. 3. Amplitude dependences of (a) decrement and (b) YMD for Zn crystal at frequency 0.5 Hz.

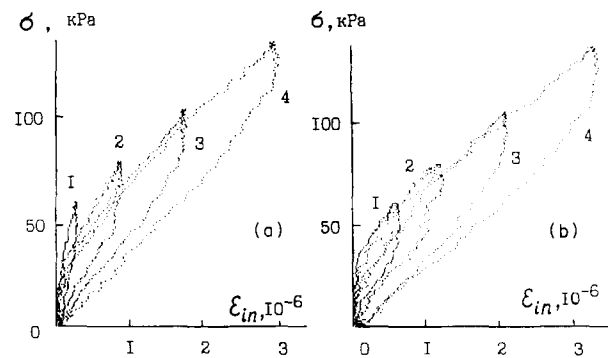


Fig. 4. Stress–inelastic strain HLs for Zn crystal at frequency 0.5 Hz and stress amplitudes 30 (1), 39 (2), 50 (3) and 65 kPa (4) for (a) increasing and (b) decreasing stress amplitudes.

are absent and the defect structure is stable (see Fig. 2a for Al crystals and data of refs. 1 and 2 for NaCl crystals).

### 3. Discussion

All the peculiarities observed in Al and Zn samples can be easily and self-consistently explained by taking into account the concept of effective and internal stress hysteresis contributing to the ADIF. The HL symmetry (Figs. 2a and 4) shows that the inelastic strain at low frequencies is not controlled by dislocation breakaway from pinning PDs. On the other hand, bias stress experiments at frequencies near 10 MHz [4, 5] revealed in 5N Al and Mg changes in high frequency sound absorption with static bias stress and a redistribution of PDs in dislocation glide planes at room temperature.

In our experiments the influence of static stress on the ADIF and the time dependence during the measurements of amplitude dependences in Al were not observed. This means that the amplitude-dependent inelastic strain in Al in our experiments, in contrast with the amplitude-independent strain at high frequencies, is not controlled by dislocation loop bowing and changes in mean loop length. This conclusion seems to be correct also at 100 kHz, where amplitude hysteresis is observed only in the amplitude-independent background [3]. As for alkali halides, the mechanism controlling the inelastic strain at low frequencies and high stress amplitudes is dislocation interaction, resulting in IS field hysteresis. This is further confirmed by the weak and decreasing frequency dependence of the ADIF. Such behaviour is similar to that observed in LiF [2] and typical of the case where the thermally activated frequency maximum lies at relatively high frequencies ( $10^2$ – $10^4$  Hz) and the contribution of  $\sigma^*$  to the ADIF in the infrasonic range is rather low. The quantitative separation of the different mechanisms contributing to the ADIF in Al, performed in accordance with the technique of ref. 2 and taking into account the quasi-static, thermally activated and athermal components of the ADIF, will be given in ref. 3.

Data on the ADIF and inelastic strain in Zn under steady state conditions are also in accordance with the concept of  $\sigma^*$  and  $\sigma_i$  contributing to the ADIF. It was shown in ref. 2 that the contribution of dislocation–PD interaction increases with decreasing stress amplitude. The hysteresis of the ADIF and YMD for increasing and decreasing amplitudes observed in Zn single crystals at low oscillatory stresses can be explained by the redistribution of pinning PDs which are mobile at room temperature. This effect is most pronounced at low stresses and vanishes with increasing stress amplitude, when  $\sigma_i$  hysteresis becomes predominant (see Figs. 3

and 4). The dislocation pinning and PD redistribution are most clearly visible for the initial loading cycles after the pause at constant stress. The HLs obtained for the first 4 cycles are shown in Fig. 5. The sample was kept at constant compressive stress at point 1. Then a sinusoidal compressive starting stress with an amplitude of 60 kPa was superimposed on the static stress. Only a relatively small amount of strain was accumulated during the first quarter of a cycle until point 2. Then a step-like increase in inelastic strain occurs near the maximum value of applied stress, thus forming an HL of the breakaway or Granato–Lücke type. The changes in the HL shape during the second and fourth cycles of loading correspond to the redistribution of the pinning environment shown schematically in Figs. 5d–5f.

For stress amplitudes lower than those shown in Fig. 5, an ordinary HL of the unlocalized friction type is formed even in the first cycle of loading. This means that at low stress amplitudes dislocations are moving inside the pinning environment and a redistribution of the PD density occurs even without a breakaway of dislocations. No breakaway is observed also at lower frequencies, when the PD mobility is high enough to be swept by mobile dislocations. According to the models of PD redistribution [4], the area with excessive PD concentration has to be formed in this case at strains near the tops of the HLs.

A special experiment was performed to obtain this PD distribution correctly. For this purpose the sample was subjected to 2 cycles of loading at a stress amplitude of 31 kPa and a frequency of 0.02 Hz. Curve 1 in Fig. 6a shows the second cycle. Then the stress amplitude was immediately increased to 43 kPa and again 2 cycles were stored for the new amplitude. One can clearly see the regions (A, B) near the vertices of HL 1 where

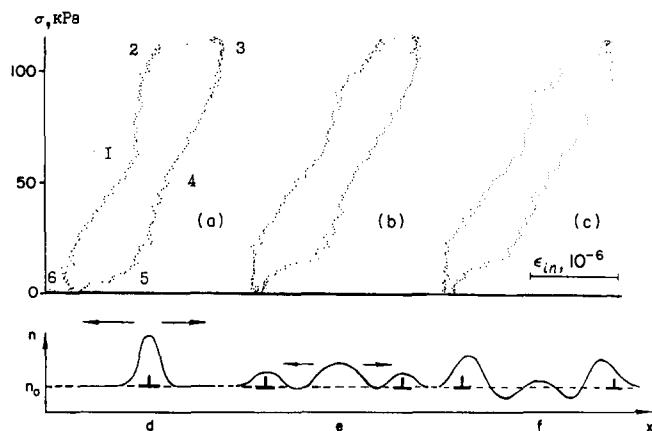


Fig. 5. Stress–inelastic strain HLs for Zn crystal at frequency 0.2 Hz and stress amplitude 60 kPa obtained after pause of 20 s for (a) first, (b) second and (c) fourth cycles of loading; d–f, corresponding schematic representation of PD redistribution in glide planes of mobile dislocations.

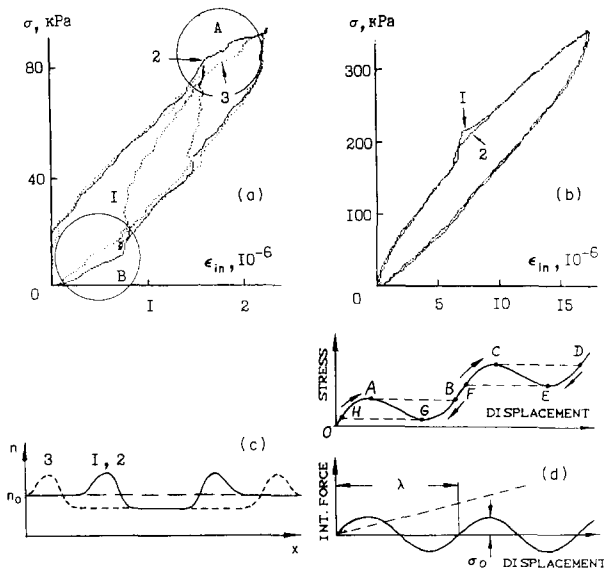


Fig. 6. (a, b) Stress–inelastic strain HLs for Zn crystal with corresponding schematic representation of (c) PD sweeping by mobile dislocations and (d) mechanism of internal stress field hysteresis: a, stress amplitudes 31 (1) and 43 kPa (2, 3), frequency 0.02 Hz; b, stress amplitude 170 kPa, frequency 0.5 Hz.

additional stress must be applied in the first cycle (HL 2) to provide the same strain as in the second cycle (HL 3 and scheme in Fig. 6c). This inhomogeneous PD distribution is already removed considerably by the second cycle of loading. This fact indicates a rather high mobility of PDs, which causes some anomalies in the frequency spectra of the ADIF and YMD and the appearance of “jerky” strain at frequencies lower than approximately 0.02 Hz [6].

To study the origin of the IS hysteresis data for Zn single crystal was used. The sample was loaded with a rather high stress amplitude of 160 kPa after a pause of 20 s which was necessary to pin the dislocations and thus to create the area with excessive PD concentration. Two HLs registered in the first 2 cycles of loading are shown in Fig. 6b. One can see that the stress which is necessary to break away the dislocations from the pinning environment during the first loading cycle can be estimated as 50 kPa (see curve 1) in accordance with the data in Fig. 5a. The anomaly in the middle part of the HL is clearly visible in the second cycle, but it became more diffuse in the strain range and lower in the value of the breakaway stress. The most remarkable fact is that no peculiarities are observed in the unloading part of the HL even for the first cycle.

This experiment demonstrates that the same regions of glide planes are overcome by dislocations in different manners when the strain becomes considerable. However, at low strain amplitudes the same pinning environment is revealed both during loading and during unloading of the sample (see Fig. 6a). On the other

hand, we concluded that for large strain amplitudes the IS hysteresis is dominant in the ADIF. A general approach predicting the formation of HLs of the unlocalized friction type owing to dislocations overcoming “ripples” in IS fields was put forward by Kressel and Brown [7]. This model is shown schematically in Fig. 6d. Two scales of IS fields are essential: a short-range scale with stress amplitude  $\sigma_0$  and period  $\lambda$  (solid curve) and a long-range scale which is assumed to be linear (dashed line). These two components give the total IS profile represented by the solid curve in the upper part of Fig. 6d. The dislocation hysteresis during the overcoming of the IS is represented by 0–A–B–C–D for forward movement and D–E–F–G–H–0 for backward movement. Dislocations are moving in regions AB, CD, EF and GH under the action of an unbalanced force with a high velocity limited by viscous forces.

It is evident from this model that various parts of the IS profile are overcome under the action of an unbalanced force, namely AGB and CED during loading and ECF and GAH during unloading, and only one part of the IS profile (BF in Fig. 6d), which is overcome under a balanced force when the velocity of dislocation movement is determined by the applied stress rate, coincides for loading and unloading. It is also evident that the higher the amplitude of the IS “ripples”,  $\sigma_0$ , the less is this region BF overlapping for dislocation movement in opposite directions. At some value of  $\sigma_0$ , dislocations will move under the action of a balanced force during loading and unloading in totally different parts of the glide plane. Supposing that there is a continuous spectrum of “ripple” amplitudes  $\sigma_0$ , we have to conclude that if we create some regions with excessive PD concentration during loading of the sample, *e.g.* somewhere on part FC of the strain–displacement hysteresis, then this “marked” part of the glide plane will be overcome under the action of an unbalanced force during unloading when the strain amplitude becomes high enough.

The above analysis shows that in real structures different regions of dislocation glide planes together with PDs are overcome during cyclic movement of dislocations under the action of an unbalanced force owing to dislocation interaction. This is why ADIF models of the unlocalized friction type, based on the concept of dislocations overcoming a two-dimensional random array of PDs [8], are not valid for high inelastic strain amplitudes.

The origin of the IS “ripples” may be (i) contactless collective action of a large number of dislocations or (ii) contact interaction with a single dislocation. The probability of the contribution of each of these mechanisms to IS hysteresis for different crystals and strain amplitudes will be discussed in ref. 6.

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