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#### **RESEARCH NOTE**

## Internal friction in indium single crystals

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**Abstract.** Low frequency, internal friction measurements made as a function of temperature on pure indium in single crystal form have shown three effects which could be resolved into the following three separate mechanisms: (i) A frequency insensitive peak in internal friction observed at about 170 K is believed to be associated with the motion of twin boundaries; (ii) A relaxation measured at about 273 K is interpreted as originating from the motion of dislocations interacting with vacancies; (iii) A background relaxation between 320 K and the melting point is attributed to dislocation climb.

#### 1. Introduction

This work cites three internal friction effects recently observed in 99.9999% indium single crystals during an investigation of anelastic effects in In–Tl alloys (de Morton 1969). Low frequency measurements (about 1 Hz) were made on wire specimens in an evacuated inverted torsional pendulum which eliminated tensile creep at temperatures near the melting point (429 K). Further experimental details are given by de Morton (1969). Initial measurements, made at a surface shear strain of  $4 \times 10^{-6}$ , showed two frequency dependent effects :

(i) a peak at about 273 K (subsequently referred to as the 273 K peak), and

(ii) an exponential rise in the background damping above 320 K, as shown in figure 1.



Figure 1. Internal friction of pure indium as a function of strain amplitude and temperature.  $\bigcirc$ ,  $\bullet$ : strain amplitude = 3 × 10<sup>-5</sup>, frequency = 0.5, 1.4 Hz respectively.  $\triangle$ ,  $\blacktriangle$ : strain amplitude = 4 × 10<sup>-6</sup>, frequency = 0.3, 1.7 Hz respectively.

On increasing the strain amplitude to  $3 \times 10^{-5}$  an additional peak was observed at about 170 K (subsequently referred to as the 170 K peak) (figure 1) which was insensitive to

frequency; the overall background damping also increased without affecting the character of the two effects already mentioned. These three effects will now be discussed separately.

#### 2. Peak at 170 K

The fact that the peak at 170 K was frequency insensitive (ie not thermally activated) and that it was observed in a monocrystal of high purity eliminates Bordoni, Zener and grain boundary type relaxations as possible explanations for this effect. Indium solidifies as a face centred tetragonal structure (c/a 1.075) which in single crystal form is lightly twinned. The presence of twins suggests a possible explanation for this peak, since it is known that the migration of twin boundaries can give rise to internal friction (Zener 1948). Furthermore, it has been demonstrated (Remant *et al.* 1964) by electron microscopy on thin films of indium that, below 188 K, fine twins ( $0.1-0.2 \mu m$  in width) are nucleated by the movement of dislocations in the films. The dislocations themselves are generated by the combined effects of the electron irradiation and stresses associated with the contaminated layer which formed on the specimen during examination in the microscope. As this layer increases in thickness the twins grow, due to the associated increase in stress, whilst heating the film above 188 K causes the twins to shrink and disappear.

These observations, and the increase in internal friction measured at the higher strain amplitude, prompt the suggestion that dislocations are generated in the internal friction specimen at low temperatures by the stress of measurement, and similarly, these dislocations nucleate fine twins. The internal peak at 170 K could then be accounted for by the reversible stress-assisted motion of twin boundaries producing a reorientation of tetragonal regions in the volume swept by the twin interfaces. At low temperatures (less than 150 K), the stress would be insufficient to propagate twin boundaries since the stress to maintain a given velocity of twin propagation increases with decreasing temperature (Cooper and Washburn 1967) and damping would therefore decrease. At higher temperatures (greater than 200 K), the reduction of micro-twins on heating above 188 K, observed by Remant et al., would also diminish damping. On this basis, a shear stress  $\sigma$  for the propagation of twins in indium can be estimated, since  $\sigma = \epsilon G$ , where G is the shear modulus  $(0.65 \times 10^6 \text{ g mm}^{-2}; 0.64 \times 10^{10} \text{ N m}^{-2})$  and  $\epsilon$  the shear strain  $(3 \times 10^{-5})$ . This gives a value of 20 g mm<sup>-2</sup> ( $1.96 \times 10^5$  N m<sup>-2</sup>) for twin propagation in this indium single crystal at 170 K; the shear stress for twin propagation in zinc single crystals at 300 K is  $150-450 \text{ g mm}^{-2}$  (1·47 × 10<sup>6</sup>-4·4 × 10<sup>6</sup> N m<sup>-2</sup>) (Cooper and Washburn 1967).

#### 3. Relaxation at 273 K

The activation energy  $Q_{\rm R}$  and frequency factor  $\tau_0$  for this relaxation were estimated from the peak shift with frequency, and are shown in table 1. The activation energy and

# Table 1. Values of activation energy $Q_R$ , frequency factor $\tau_0$ , and relaxation strength $\Delta G$ at different strain amplitudes, for the relaxation at 273 K

Strain amplitude	$Q_{ m R}\left({ m eV} ight)$	$ au_{0}$ (s)	$\Delta G$
$4 \times 10^{-6}$	$0.49 \pm 0.09$	$5 \times 10^{-11}$	0.032
$3 \times 10^{-5}$	$0.52 \pm 0.09$	$3 \times 10^{-11}$	0.095

frequency factor values were essentially independent of strain amplitude, whereas the relaxation strength  $\Delta G$  increased by a factor of three when the strain amplitude was increased from  $4 \times 10^{-6}$  to  $3 \times 10^{-5}$ . Measurements of the half peak width of this relaxation peak were also within a few per cent of the theoretical half peak width, indicating a relatively simple activated process. Comparison of  $Q_{\rm R}$  with the activation energy for self-diffusion  $Q_{\rm D}$ , which is 0.78 eV (Eckert and Drickamer 1952), showed no correlation. In seeking an explanation for this relaxation, the marked strain-amplitude dependence of internal friction and the unusually high value of  $\tau_0$  (5 × 10<sup>-11</sup> s) is worth noting. Similar high  $\tau_0$ 

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results, for anelastic relaxations on deformed copper and gold, led Okuda and Hasiguti (1963) to consider a relaxation mechanism involving the association of dislocations with vacancies and vacancy complexes. Turner and De Batist (1964) subsequently showed that Okuda and Hasiguti's data presented on a Wert and Marx (1953) type plot gave a straight line with a characteristic slope. The present data also fit closely on this line.

The present results, however, are equilibrium relaxations observed on well annealed single crystals at 0.65  $T_{\rm m}$  on both heating and cooling. It is suggested that existing dislocations are activated during the measurement and, at an optimum temperature, interact with thermally created defects to produce a viscous drag on the dislocations. Assuming that the most probable lattice defects available at these temperatures to interact with dislocations are vacancies, the activation energy measured should be that for vacancy migration,  $Q_{\rm M}$ . A value of 0.49 eV for  $Q_{\rm M}$  in indium is, however, somewhat higher than expected; for example, in fcc metals  $Q_{\rm M}/Q_{\rm D}$  is 0.36–0.48 (Cottrell 1964) whereas  $Q_{\rm R}/Q_{\rm D}$ for the current work is 0.6. However, the value of  $Q_{\rm R}$  measured could conceivably be the sum of  $Q_{\rm M}$  and the binding energy of a vacancy to a dislocation  $E_{\rm B}$ , by analogy with the cold work peaks observed in bcc metals (Shoeck and Mondino 1963). In this case, taking an average value of  $Q_{\rm M}/Q_{\rm D}$  as 0.42 gives  $E_{\rm B}$  equal to 0.16 eV.

#### 4. Background relaxation

Above 320 K (0.75  $T_{\rm m}$ ), internal friction increases rapidly up to the melting point and, as shown in figure 1, the logarithmic plot is linear with 1/T (K). The frequency dependence of this background damping, which is observed in both single and polycrystalline specimens, has suggested a relaxation process of the following relationship (Niblett and Wilks 1960):

$$Q^{-1} = \delta/\pi = A \exp(-Q_{\rm B}/kT).$$
 (1)

Estimates of the activation energy  $Q_{\rm B}$  from the slopes of the curves shown in figure 1 give values of 0.24–0.28 eV. These values are about 0.3  $Q_{\rm D}$ . Schoeck *et al.* (1964) pointed out that the magnitude of background internal friction is too large to be explained solely by a mechanism involving point defects, and suggested that it is probably associated with a thermally activated dislocation relaxation. These workers also questioned the validity of equation (1), and derived the following expression for the background relaxation:

$$Q^{-1} = K\{\omega \exp(Q_{\rm B}^*/kT)\}^{-n}$$
<sup>(2)</sup>

where k and n are constants,  $Q_{\rm B}^*$  is the true activation energy for the viscous process and  $\omega$  is the oscillating frequency. The 'apparent' activation energy  $Q_{\rm B}$  is related to  $Q_{\rm B}^*$  by

$$Q_{\rm B} = n Q_{\rm B}^*. \tag{3}$$

Since *n* is characteristically less than unity,  $Q_{\rm B}^*$  is always greater than  $Q_{\rm B}$ ; for Al it is approximately equal to  $Q_{\rm D}$ . The frequency dependence of  $Q^{-1}$ , indicated in equation (2) and shown in figure 1, has been used to obtain values of *n* by plotting  $\ln Q^{-1}$  against  $\ln \omega$ . The value of *n* obtained was 0.4 which, through equation (3), gives 0.65 eV for  $Q_{\rm B}^*$ . Since this is reasonably close to  $Q_{\rm D}$ , the rate determining process for this background relaxation is probably dislocation climb. If this is so, the technique would be of value in determining the creep behaviour in materials near their melting points.

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