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Estimation of binding energy between a dislocation and vacancy and concentration of vacancies from ultrasonic attenuation measurements

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Abstract. Cottrell's binding energy between a dislocation and vacancy and concentration of vacancies has been calculated using ultrasonic attenuation measurements in single crystals of pure tin at 300K for frequencies varying from 3 to 50 MHz and at different dislocation densities.

Keywords. Ultrasonic attenuation; dislocation; vacancy; Cottrell's binding energy; Sn.

1. Introduction

The measurements (Chaudhuri and Verma 1978) of ultrasonic attenuation in single crystals of pure tin at 300 K for frequencies varying from 3–50 MHz exhibit resonance type of behaviour in the attenuation which arises due to dislocations only, excluding the contribution due to electrons which is negligible at room temperature. Granato and Lücke (1956) have drawn the analogy between the vibration under an alternating stress of a dislocation line segment pinned down through Cottrell (1948) mechanism by point defects *i.e.* vacancies or interstitial atoms, substitutional or interstitial impurity atoms and the problem of forced damped vibration of a string. The resonance frequency is given by

$$\omega_0 = 2\pi \nu_0 = \frac{1}{L_c}\left(\frac{2G}{\rho\,(1-\sigma)}\right)^{1/2}, \tag{1}$$

where G is the shear modulus of the material, ρ is the density of the material and σ is the Poisson's ratio. The length L_c is determined by the minor pins due to point defects. In our experimental crystal of high purity tin (99·9999%) the point defects are mainly vacancies and act as minor pins. It is assumed that for zero applied stress the dislocations are straight and pinned down by the vacancies already present in the crystal. The interaction between vacancy and dislocation causes the vacancies to segregate round dislocations in equilibrium distributions. Each dislocation gathers round itself an 'atmosphere' of vacancies, the equilibrium concentration (Granato and Lücke 1966) of which, at a point is

$$C = C_0 \exp\,(Q/kT), \tag{2}$$

where C_0 is the average concentration of vacancies in the lattice and $C = n/N$, n indicates the number of vacancies along a dislocation line and N indicates the number of atomic lengths of dislocation line in the specimen. In general the concentration C of vacancies on the dislocation line is larger than the overall concentration of vacancies C_0 in the lattice. In (2) Q is the interaction energy between a dislocation and a vacancy, a is the atomic distance along the dislocation line, k is the Boltzmann's constant and T is the temperature.

By knowing ν_0, L_c can be found using (1) and thus C can be calculated. Since C_0 can be estimated at room temperature T, for our annealed specimen, by utilizing the relation

$$C_0 = A \exp(-U_{fv}/kT), \tag{3}$$

where U_{fv} is the energy formation of the vacancy and A a constant of the order of unity; one can calculate Q using (2). Moreover, experimentally it has been observed by Chaudhuri and Das (1976) that the resonance frequency ν_0 increases with an increase in dislocation density and as a result L_c decreases. The concentration C of the vacancies is given (Granato and Lücke 1966) by a/L_c and consequently it increases with dislocation density. Overall concentration of vacancies C_0 also increases with C as both are related through (2) where Q is constant. Thus ultrasonic attenuation measurements can be utilized for the estimation of both C and C_0.

2. Experiments

A conventional double ended ultrasonic pulse echo technique was used to measure the attenuation of longitudinal ultrasound of frequencies lying in the range 3–50 MHz. The pulse amplitude was kept fixed at 100 V peak to peak. The preparation of the single crystal of tin of high purity (99·9999%) and other details have already been reported by Chaudhuri and Verma (1978). Measurements have been taken along the [100] direction under different conditions of strain. By etching such specimens with the help of a 50% $FeCl_3$ solution, etch pits at the sites of dislocations were revealed and thus the dislocation densities corresponding to any physical state of specimens were estimated. The etch pits were observed under a phase contrast projection microscope. The dislocation densities were gradually reduced by annealing the specimen under controlled conditions for progressively longer periods of time.

3. Experimental results and discussion

Figure 1 gives the results of the ultrasonic attenuation measurements at 300K at various frequencies for different physical states of the specimen. It has been shown (Chaudhuri and Verma 1978) that the attenuation that is measured at 300K can be taken as entirely due to dislocations, the contribution due to electrons being practically absent. It will be noticed that there is a resonant behaviour in the attenuation due to dislocation. The set of curves corresponding to different dislocation densities shows the occurrence of resonance peaks. It will be seen that ν_o increases with increase in dislocation density. The resonance frequency

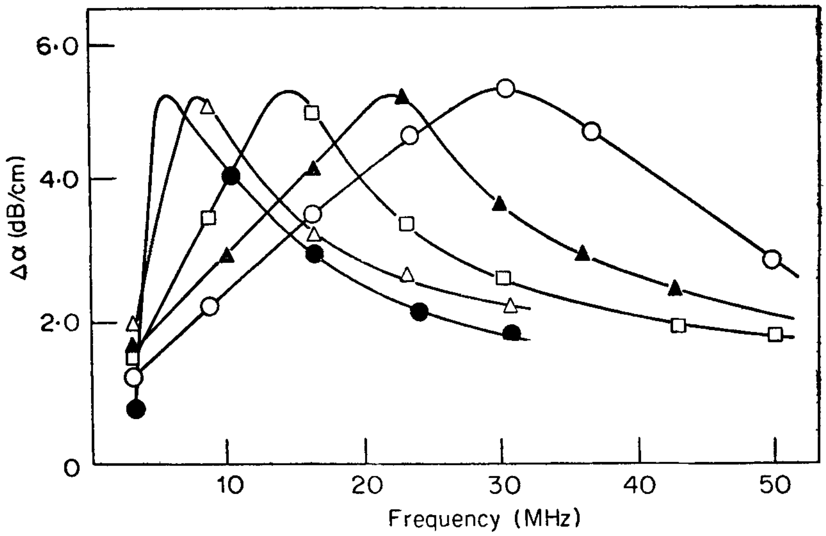

Figure 1. Frequency dependence of $\Delta\alpha$ at different dislocation densities Λ (Λ's in 10^5 cm^{-2} are given in brackets) ● (0.01); △ (0.06); □ (2.00); ▲ (20,00); ○ (110.00).

ν_0 gives the average loop length L_c and hence the concentration of pinning points *i.e.* vacancies along the dislocation line.

In our experimental crystal of pure tin in highly annealed state (dislocation density $\Lambda = 0{\cdot}01 \times 10^5\,\mathrm{cm}^{-2}$) $C_0 = 38{\cdot}3 \times 10^{-10}$ at temperature $T = 300\mathrm{K}$ using $U_{f_v} = 0{\cdot}5$ eV for tin (Friedel 1964).

For a highly annealed state, the resonance frequency was found to be 5·5 MHz and consequently $L_c = 0{\cdot}0072$ cm, $C = a/L_c = 138{\cdot}9 \times 10^{-8}$ taking $a = 10^{-8}$ cm. Substituting these two values of C and C_0 in (2) and substituting for $T = 300\mathrm{K}$, it has been found that the Cottrell binding energy $Q = 0{\cdot}15$ eV. Bullough and Newman (1962) have also reported that vacancies interact elastically with a dislocation. A dilational strain field is formed round a vacancy and hence a vacancy is attracted to the compressive region above the slip plane of an edge dislocation. According to them

$$Q = -\frac{15\,VGb^2}{4\pi^2(1-\sigma)(7-5\sigma)R^2}\left[1 - \frac{(1+6\sigma-5\sigma^2)\sin^2\theta}{5}\right], \tag{4}$$

where G is the shear modulus, σ is the Poisson's ratio, V is the atomic volume, R is the distance between the vacancy and the dislocation and θ is the angle between R and the Burgers' vector b.

Using the calculated value for Q in (2), C_0 is calculated for different physical states of the specimen as C is known for any particular physical state from ultrasonic attenuation measurements. Table 1 illustrates how with increase in dislocation density ν_0, L_c, C and C_0 change ($G = 1{\cdot}5 \times 10^{11}$ dyne cm^{-2}, $\sigma = 0{\cdot}33$, $a = 10^{-8}$ cm, $\rho = 7.3$ gm/cc, $Q = 0{\cdot}15$ eV for the tin sample used.).

The table clearly shows that with increase in dislocation density ν_0 increases and as a result L_c decreases. It also shows that C and C_0 increase with dislocation density. This means that the dislocations behave as sources for vacancies. This results in an

Table 1. Dependence of concentration of vacancies on dislocation density

$\Lambda \times 10^{-5}$ cm^{-2}	ν_0 MHz	L_c cm	$C = a/L_c$ $C \times 10^8$	$C_0 \times 10^{10}$
0·01	5·5	0·0072	138·9	38·3
0·06	8·0	0·0049	204·1	56·4
2·00	14·5	0·0027	370·3	102·3
20·00	22·5	0·0018	555·5	153·4
110·00	30·0	0·0013	769·3	212·6

increase in the number of minor pins and consequently L_c, the average distance between the minor pins decreases.

These measurements of ultrasonic attenuation, therefore, clearly show that dislocations interact with vacancies and the value of $Q = 0{\cdot}15$ eV in the tin sample used. These results also show that more vacancies are created in the sample due to increase in the dislocation density and dislocations play a central role in bringing the vacancy concentration in the crystal to thermodynamic equilibrium *via* the Cottrell interaction.

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