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The interaction of point defects with dislocations in high-purity silver above room temperature

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Abstract. Damping and modulus measurements were made on high-purity, polycrystalline silver during 1.5 MeV electron irradiations in the temperature range 310 K to 590 K. A suggested interpretation of the pinning rate data gave a value of 0.24 ± 0.01 eV for the difference in interstitial migration energies in the lattice and the dislocation line and an approximate value of 0.25 eV for the binding energy of a vacancy to the dislocation line. An initial increase in the decrement (peaking effect) at the onset of radiation was observed at different amplitudes and at temperatures as high as 590 K.

1. Introduction

Internal friction and Young's modulus experiments are a sensitive technique to study the interaction of point defects with dislocations undergoing an oscillatory shear stress. These experiments are usually interpreted in terms of the well known vibrating string model proposed by Koehler (1952) and refined by Granato and Lücke (1956), the KGL theory. In this theory, the motion of a dislocation line in a solid is analogous to a damped vibrating string which is pinned at both ends. The effect of electron irradiation is to produce point defects in the lattice which migrate to the dislocation line, forming nodes on the 'string'. This shortening of the average free length of the line is called dislocation pinning. Simpson and Sosin (1972) have modified the KGL theory for low frequencies by introducing the concept of defect dragging. This study was undertaken to extend the work done on copper and gold at room temperatures to silver. A series of electron irradiations were made at 20 K intervals over a temperature range of 310 K to 590 K. These were dynamic with the irradiations and data collection performed at the temperatures of interest. The state of dislocation pinning was monitored by measuring the internal friction and the modulus defect. Flux and amplitude dependence of the modulus and internal friction were also studied. The material used was high-purity, polycrystalline silver.

2. Experimental procedure

The sample was fabricated from a high-purity (99.999%), polycrystalline, silver rod 1.27 cm in diameter obtained from Alpha Products. This was machined and hand and

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chemically polished into the form of a cantilevered beam 0.71 cm long, 0.64 cm wide, and 0.011 cm thick. After mounting the sample on one-half of a parallel plate capacitor, a capacitative drive and pick up system was used to control its frequency of vibration and amplitude. The sample was driven at its resonance frequency of approximately 900 Hz and at a constant preset amplitude. The drive voltage, V, applied to the sample to maintain constant amplitude, and the period of vibration, τ, were continuously monitored and recorded. The details of this data acquisition system are given elsewhere (Simpson and Sosin 1977). The drive voltage and period can be shown (Simpson and Sosin 1977) to be related to the logarithmic decrement, δ, and Young's modulus, Y, by the equations

$$\delta/\delta_0 = (V/V_0)^2(\tau/\tau_0)^2 \tag{1}$$

$$Y = k/\tau^2 \tag{2}$$

where k is a proportionality constant and the subscript $_0$ denotes pre-irradiation values.

The electron irradiations were performed using the 2.5 MeV Van de Graaff accelerator at Wright State University. A description of the beam and sample geometries have been detailed elsewhere (Goldstone *et al* 1980). Before each irradiation the sample was annealed at 650 K for 20 min to remove any damage caused by the previous irradiation. This procedure always produced the same relative pre-irradiation values of internal friction and modulus. After annealing, the sample was slowly cooled to the selected temperature.

The sample was irradiated with 1.5 MeV electrons at fluxes ranging from 3.1×10^{11} to 3.1×10^{13} electrons $cm^{-2}\,s^{-1}$; typically a flux of 1.54×10^{12} electrons $cm^{-2}\,s^{-1}$ was used. The current was measured by collection in a Faraday cage behind the sample.

The number of pinning points, n_d, added to dislocation lines can be shown to be linear with time with the assumptions of a constant production rate of interstitials and vacancies, high temperature (i.e. above 300 K), and diffusion of interstitials along dislocation lines to nodal point traps (Simpson *et al* 1972). Then n_d can be related to the changes in Young's modulus during irradiation (Simpson *et al* 1972). Since $Y = k/\tau^2$, then

$$n_d \propto (\Delta Y/Y)_0/(\Delta Y/Y) - 1 = (\tau_0^2 - \tau^2)/(\tau^2 - \tau_e^2) = N_{dy} \tag{3}$$

where $\Delta Y = Y_e - Y$ with Y_e being the elastic (fully pinned) value and N_{dy} is the inverse normalised modulus minus one.

3. Results and discussion

3.1. The peaking effect

A series of irradiation runs were made at temperatures ranging from 310 K to 590 K in 20 K increments. A 1.5 MeV electron beam with a flux of 1.54×10^{12} electrons $cm^{-2}\,s^{-1}$ was used for approximately 70 min for each run. For the sake of clarity not all of these runs are shown on the graphs.

A semilog plot of N_{dy} versus time of irradiation for the runs from 310 K to 410 K in figure 1 shows the number of pinners increasing rapidly in the beginning and then levelling off toward a saturated value. This equilibrium is reached when there are as many pinners arriving at the dislocation line as are leaving it. Pinners are thermally activated and will arrive at the line more quickly at higher temperatures. Thus the initial

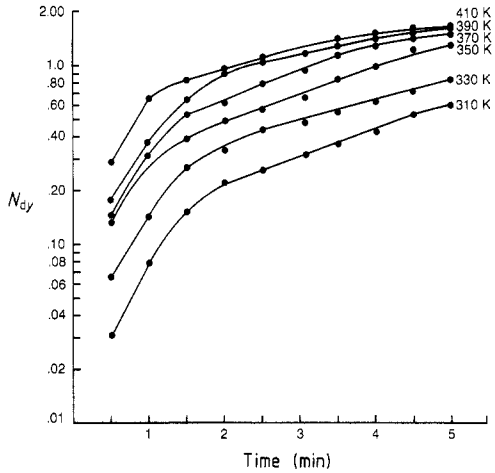

Figure 1. The normalised modulus defect minus one (N_{dy}) versus irradiation time at different temperatures.

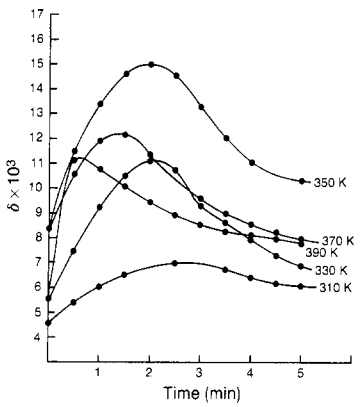

Figure 2. The log decrement versus irradiation time at different temperatures.

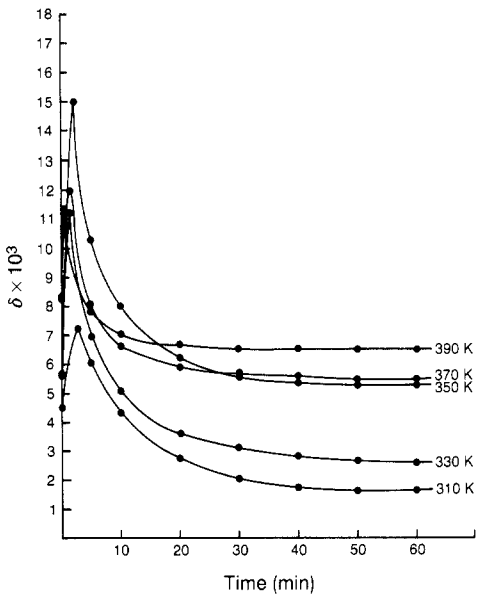

Figure 3. The log decrement versus irradiation time at different temperatures.

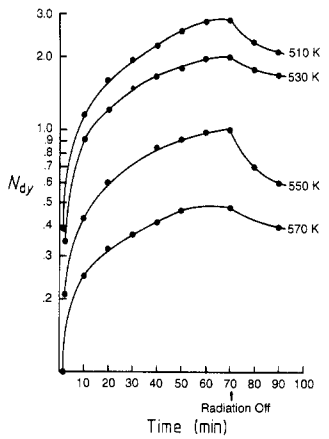

Figure 4. The normalised modulus defect minus one (N_{dy}) versus irradiation time at different temperatures.

value of N_{dy} increases with temperature and saturation is reached sooner with higher temperatures.

A plot of the decrement data for the runs from 310 K to 390 K (figure 2) displays the peaking effect. Note that the peak is a maximum at 350 K. Figure 3 shows the data for

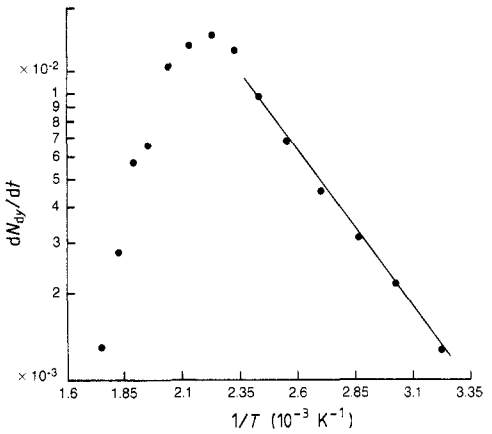

Figure 5. The initial pinning rate versus the inverse of temperature.

the same runs as they reach saturation. It is evident that the saturated value increases with increasing temperature. This is because the background internal friction, regardless of the pinning mechanism, increases with temperature.

When the radiation is turned off, defects are no longer added to the lattice. The dislocations can experience depinning. This is evident at the higher temperatures when the defects migrate more readily. Figure 4 illustrates the depinning graphically. Note that the greatest rate of depinning occurs for the 550 K run.

3.2. Activation energy analysis

A semilog plot of the initial pinning rates versus $1/T$ in figure 5 is approximately linear in the low temperature region. The following equation relates the pinning rate, before significant depinning occurs, to temperature (Goldstone *et al* 1980):

$$dN_{dy}/dt = (PlD_0t/K_0)\exp[-(H_m - H_d)/kT] \tag{4}$$

where P is the defect production rate, l is the dislocation line length, D_0 and K_0 are the diffusion constants to and along a dislocation, respectively, H_m is the migration enthalpy of a defect due to the dislocation line, and H_d is the migration enthalpy of a defect along the dislocation line. The linear portion of the curve yields a difference in migration energies in the lattice and the dislocation line $(H_m - H_d)$ of 0.24 ± 0.01 eV. This compared to 0.18 eV found by Goldstone *et al* (1980) for copper and 0.07 eV found by Brown (1977) for gold.

If we can assume that the initial pinning is due primarily to interstitials, which is indicated by quenching and irradiation experiments on copper (Simpson and Kerkhoff 1976), then the difference in migration energies quoted would be for interstitials.

A semilog plot of N_{dy} at saturated values versus $1/T$ in figure 6 is approximately linear in the high-temperature region. The number of pinners at saturation can be related to temperature by the following equation (Simpson and Kerkhoff 1976).

$$n_d \propto Pl\exp(-E_a/kT) \tag{5}$$

where E_a is the activation energy for a point defect along a dislocation line. The slope of the linear portion of the curve yields an activation energy of 0.90 ± 0.03 eV. If we

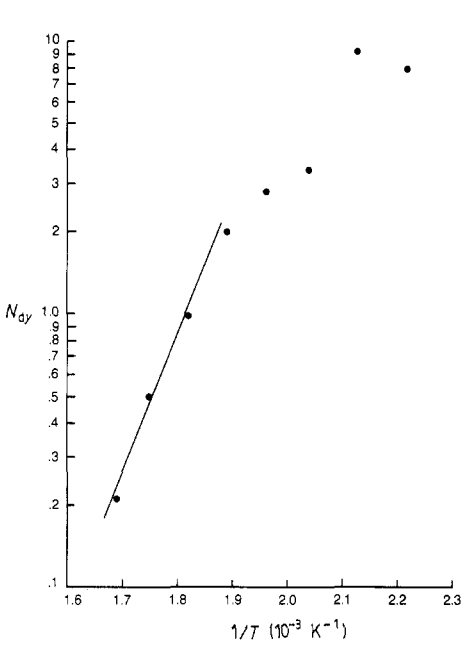

Figure 6. The saturated value of the normalised modulus defect minus one (N_{dy}) versus the inverse of temperature.

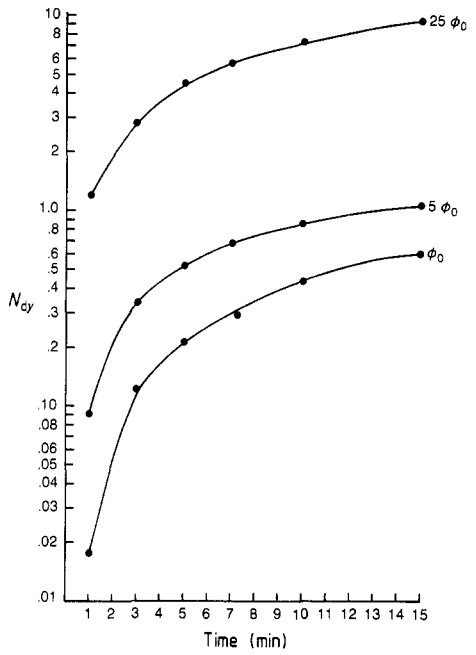

Figure 7. The normalised modulus defect minus one (N_{dy}) versus irradiation time for different electron doses at 350 K. $\phi_0 = 3.1 \times 10^{11}$ electrons $cm^{-2}\,s^{-1}$.

assume that vacancies are primarily the point defects responsible for pinning at high temperatures, then E_a consists of the binding energy of the vacancy, E_b, to the dislocation plus the migration energy of the vacancy, E_m, through the lattice. Taking E_m to be 0.64 eV as reported by Balluffi (1978) yields a value of $E_b \simeq 0.25$ eV which is in good agreement with the binding energy for a copper vacancy of 0.25 eV as reported by Simpson and Kerkhoff (1976) and in reasonable agreement with the theoretical value of 0.2 eV given by Kuhlmann-Wilsdorf (1965).

3.3. Varying dose runs

Runs at 350 K were made at fluxes of $\phi_0 = 3.1 \times 10^{11}$ electrons $cm^{-2}\,s^{-1}$, $5\phi_0$, and $25\phi_0$. A comparison plot of N_{dy} versus time of irradiation in figure 7 shows a much more rapid increase in the number of pinners in the higher dose runs. The decrement data for the same runs in figure 8 shows that the highest dose run reaches a peak earlier and decreases much faster than the lower dose runs.

3.4. Amplitude dependence

A series of runs at 350 K were made at different amplitudes corresponding to different maximum strains. As seen in the comparison plot of the decrement values in figure 9, the peak height increases with amplitude. The decrease of the decrement is also much slower at larger amplitudes. The strain chosen for the other runs of this experiment, $\varepsilon_0 = 12 \times 10^{-8}$, exhibits here a large well defined peak. This amplitude dependence is

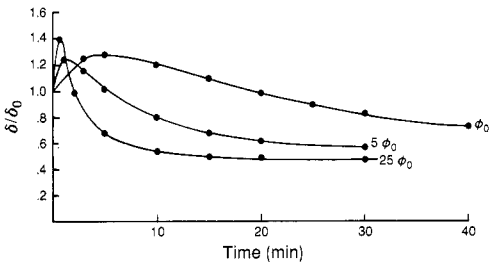

Figure 8. The normalised log decrement versus irradiation time for different electron doses at 350 K. $\phi_0 = 3.1 \times 10^{11}$ electrons $cm^{-2}\ s^{-1}$.

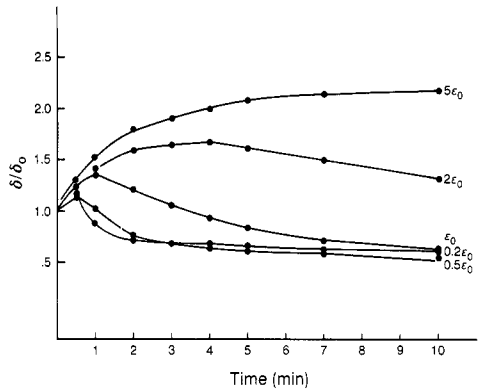

Figure 9. The normalised log decrement versus irradiation time for different maximum strains at 350 K. $\varepsilon_0 = 12 \times 10^{-8}$.

in general agreement with the work done by Kerans and Simpson (1979) who found that the decrement peak in copper increased with amplitude and dropped off more slowly at higher amplitudes. Qualitatively, the amplitude dependence of the peaking effect can be explained by the concept of defect dragging. As the amplitude of the motion of the dislocation becomes larger it then takes more defects to pin it. As each of these defects is dragged along, the internal friction becomes greater with greater amplitudes and the decrease of the decrement peak, signifying pinning, takes place much later. The effect of a high amplitude is to inhibit pinning. Esnouf and Fantozzi (1983) have developed a model that not only exhibits the peaking effect but also predicts the amplitude dependence. This model includes the effect of the dislocation line encountering defects as it sweeps through the lattice. It also predicts the disappearance of the peak altogether at a low enough amplitude. The disappearance of the peaking effect was not seen in this experiment. However the lowest strain amplitude obtainable was limited by the decreasing stability of the signal. The disappearance of the peak was also not seen in the work done in silver (at lower temperatures) by Girard and Minier (1978) but was seen in copper by Lauzier *et al* (1975).

4. Conclusions

This experiment extends the low-temperature work done in silver by Girard and Minier (1978) to the range 310–570 K. This experiment provides further evidence of the peaking effect. The initial increase in the decrement, followed by a decrease was found at temperatures ranging from 310 K to 570 K with the largest peak occurring at 350 K.

In addition, with the assumptions that interstitials are the point defects primarily responsible for pinning at low temperatures and vacancies at high temperatures, an activation energy for both interstitials and vacancies was found. By plotting the initial pinning rates at lower temperatures versus $1/T$, an activation energy (the difference in migration energies in the lattice and dislocation line) for interstitials was found to be 0.24 ± 0.01 eV and compared with that in gold and copper. The interstitials in silver were found to be more tightly bound than in gold or copper. An activation energy for

vacancies, consisting of a binding energy of the vacancy to the dislocation plus the migration energy of the vacancy through the lattice, was found by plotting the saturated value of the normalised modulus defect minus one versus $1/T$. This was found to be 0.90 ± 0.03 eV. The binding energy of the vacancy to the dislocation was then found to be approximately 0.25 eV and compared to that of copper, as well as a theoretical value for silver.

The dependence of the decrement peak with amplitude was investigated. The peak was seen at all strain amplitudes used in the experiment. The peak did not disappear as predicted by Esnouf and Fantozzi (1983). The decrement peak displayed the same general characteristics described by Kerans and Simpson (1979) in copper. However, the normal range of amplitudes obtainable here did not allow a complete comparison.

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