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FROM GAMMA IRRADIATION

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STAGE III DISLOCATION PINNING IN SILVER RESULTING
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ABSTRACT

The objectives of this work were fourfold: (1) to test the validity of the vibrating string dislocation model in silver, (2) to determine in which annealing stage long-range migration first occurs, (3) to measure the migration energy and identify the defect responsible for observed annealing, and (4) to investigate the possibility of dislocation pipe diffusion. Apparatus is described which is capable of continuously measuring changes in the elastic modulus and internal friction during gamma irradiation over the temperature range from <4 K to well above room temperature. A novel hollow-cylinder sample geometry permits use of a source strength of only 1 Ci. Survey experiments over a wide temperature range and isothermal irradiations above room temperature are discussed. The results are explained in terms of the Koehler-Granato-Lücke vibrating string dislocation model and the Thompson-Buck-Huntington-Barnes defect-dislocation interaction model.

GENERAL DESCRIPTION

We have performed experiments¹ on silver of the type pioneered by Thompson *et al.*²⁻⁴ with copper. The approach was briefly as follows: Point defects were created in high purity silver by gamma irradiations; their behavior was studied by continuously monitoring the internal friction and the elastic modulus of a specimen that was driven in mechanical vibration; the temperature was controlled in order to study the kinetics of the observed behavior; and the damage was annealed out *in situ* after each experiment so as not to disturb the dislocation structure.

APPARATUS AND SAMPLE

The apparatus design was similar to that of Thompson and Glass⁵, and is described in detail elsewhere¹. Key parameters involving the apparatus and sample are as follows:

Sample form - Hollow cylindrical, $\langle 111 \rangle$ oriented single crystal

Impurities - < 1 ppm starting, 50 ppm final, primarily Si

Dislocation density and structure - unknown, stabilized with 500°C anneals.

Mounting - Four sliding pins at midplane

Background apparatus damping - $< 1.4 \times 10^{-5}$

Source - 1 Curie ^{60}Co rod, inside sample

Flux - 3.6×10^9 gamma/(cm²-sec)

Damage rate uniformity - $\pm 25\%$ over 90% of sample volume

Temperature uncertainty - $\pm 0.25\text{K}$ during single run; $\pm 0.5\text{K}$ between runs.

Drive and detection - Electrostatic drive, FM detection, automatic phase control

Frequency - 24-27 kHz, measured to precision of 0.1 Hz.

Max. strain amplitude - 10^{-8}

Damping measurement - Free decay, resonance curve width, and drive voltage at constant amplitude. Precision - 1%.

EXPERIMENTS

We first performed survey experiments to observe the background damping and to find out in what temperature range pinning effects could be detected. We observed a broad Bordoni peak in the annealed sample prior to irradiation. It was centered at 81 K, with half maximum values at 25 K and 190 K. The decrement was 5×10^{-3} at the maximum.

After irradiation at 4 K for 24 hours with a 20-Curie source, we performed a continuous warmup. In the temperature range between 65 K and 90 K, we observed an increase in frequency of 0.06% compared to the annealed condition. This change appeared to be significant in comparison to the scatter in the data. There was no significant change in damping compared to the annealed condition in this temperature range, however. When

the sample was warmed up to room temperature and above, major changes in damping and frequency were observed, so we decided to conduct isothermal irradiations in that region. When the sample was subsequently recooled to 4 K and again warmed up, the changes were observed to persist over the whole temperature range. In particular, the damping was about 15% lower at 4 K, and about 48% lower at room temperature. The observed peak was decreased about 32% in height and shifted down in temperature to 70 K.

We also observed in other experiments that it was possible to saturate the damping change in a reasonable time, but the frequency continued to change even after long irradiation. For example, in a 75°C isothermal irradiation with a 96-Curie source, the damping stopped changing in 10 hours, while the frequency was still increasing after 23 days.

The mounting was improved to eliminate sample deformation prior to the isothermal runs. Four isothermal irradiations with the 1-Curie source were performed at the following temperatures: 24.8, 34.8, 44.0, and 53.0°C. Between irradiations the sample was annealed to return the damping and frequency to their initial values. The decrement data are shown in Fig. 1. (Note that the scales are not the same for each temperature.) At the end of the 24.8°C and 34.8°C runs, the 96-Curie source was inserted for about 150 hours. It was observed that the modulus defect change nearly doubled, with little or no concomitant change in the damping.

INTERPRETATION

The Bordoni peak⁶ has been observed by several workers. Its presence after annealing may be accounted for by deformation near the mounting pins, which had not yet been treated to promote sliding when this observation was made.

We interpret the frequency change in the range between 65 and 90 K to be due to long range migration of a point defect in Stage I. The damping change which should have accompanied it was probably below the sensitivity of our measurement. The somewhat high Stage I temperature is due to the low defect concentration and the fact that it was a continuous warmup rather than an isochronal anneal. This interpretation agrees with

the results of Keefer and Robinson⁷. The fact that damping and modulus changes which occurred near room temperature were seen to persist down to 4 K indicates that the samples were sensitive to pinning over the entire temperature range.

Since the frequency continued to change long after the damping change had saturated, the results cannot be explained by the Koehler-Granato-Lücke (KGL) vibrating string model^{8,9} if only one dislocation component is assumed. We have therefore assumed, as have others in the past¹⁰⁻¹⁴, that there are two groups. Furthermore, we have assumed that one group has a much higher average loop length than the other, so that both groups may contribute to the modulus defect, but only the group with the greater average length is responsible for the observed pinnable damping. This model was proposed but rejected by Paré and Luberman¹⁵. The irradiations with the large source at the end of the 24.5°C and 34.3°C runs suggest that the (assumed) shorter group did not contribute significantly to the damping changes observed earlier in the experiments. This enables us to interpret the damping data using the KGL model. We are not able to interpret the frequency data quantitatively because it never saturated.

In interpreting the damping data, we decided to use the Thompson-Buck-Huntington-Barnes (TBHB) defect-dislocation interaction model³ because of the similarity between our results and theirs². We did not attempt to fit the data to the Simpson and Sosin defect dragging model¹⁶ because we did not observe an initial increase in the damping, and because the vibration frequency was above the range where the dragging effect appears to dominate the pinning effect.

We did a simultaneous fit of the TBHB model to all the damping data from the four isothermal runs, using a CDC 7600 computer. It was necessary to use three transient terms in the expansion given by the TBHB model, since the higher order terms were not found to be negligible as in their work on copper. The final fit is shown by the curves in Fig. 1. The standard deviation is 1.4%, which is comparable to the precision of measurement.

The values of the parameters arrived at with this fit appear to be

reasonable. In particular, we obtained a lattice migration energy for the defect responsible for the pinning of 0.48 eV, a dislocation spacing of 10^{-3} cm, a pipe diffusion energy of 0.30 eV, and a defect-node binding energy of 0.43 eV. The value of the pre-exponential term in the lattice diffusion relaxation time and a calculation of the diffusion length from the Einstein relation indicate that the lattice diffusion is free (untrapped). The value of the pre-exponential term in the pipe diffusion relaxation time and the value of the binding energy for the nodes, indicates wide splitting of the extended dislocations, which is consistent with the low stacking fault energy of silver.

Because of the rather low value of the lattice migration energy and the fact that divacancies or di-interstitials are extremely unlikely at the low defect concentrations used, we believe some form of the single interstitial atom is responsible for the observed pinning.

CONCLUSIONS

We conclude that the results can be explained by the KGL vibrating string model if two dislocation components are assumed, one of which has a much higher average loop length than the other. The survey experiments are consistent with long-range migration in Stage I in silver. E_m is about 0.48 eV for the defect which pins dislocations in Stage III in silver, using gamma irradiation and low defect concentrations. This defect is most likely the single interstitial atom. Pipe diffusion is necessary to explain the results. The migration of point defects and their interaction with dislocations can be explained by the model of Thompson, Buck, Huntington, and Barnes.

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Fig. 1 Damping Data