

## Low-Temperature Measurements of the Young's Modulus and Internal Friction of Copper during Irradiation

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# Letters to the Editor

## Low-Temperature Measurements of the Young's Modulus and Internal Friction of Copper during Irradiation

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(Received December 20, 1956)

MEASUREMENTS of the Young's modulus and internal friction of a copper single crystal have been made during irradiation at about 21°K in the Hole 12 cryostat<sup>1</sup> in the ORNL graphite reactor with a fast neutron flux of about  $7 \times 10^{11}$  neutrons/cm<sup>2</sup> sec. The techniques of modulus and internal friction measurements were the same as reported previously.<sup>2</sup> In Fig. 1 is shown the increase in resonant frequency of the crystal with bombardment time and the accompanying decrease in internal friction. The resonant frequency and the Young's modulus  $E$  are related by

$$f = (1/2l)(E/\rho)^{1/2}, \quad (1)$$

in which  $f$  is the resonant frequency,  $l$  is the sample length, and  $\rho$  is the density. These data are consistent with those which have been obtained at room temperature at much smaller bombardments,<sup>3</sup> and can be interpreted in the same manner as dislocation pinning.

Figure 2 shows some "annealing" data which were obtained after a bombardment of  $nt=2 \times 10^{16}$ . After this period the reactor was turned off, and the crystal allowed to warm up. The solid circles show the measured resonant frequencies plotted against temperature. There is evidently a process which begins to increase the resonant frequency and offset the normal temperature decrease between 35 and 40°K. Upon recooling to 21°K, the resonant frequency was considerably higher than it was at the end of the bombardment. A second warmup indicated by the open circles again shows a knee which commences in approximately the same region and continues out to about 55°K, at which point the normal warm-up curve seems to resume. Upon recooling again

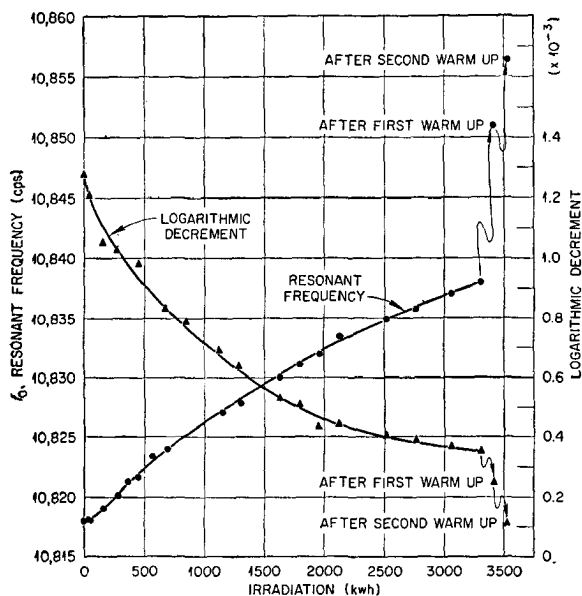


FIG. 1. Resonant frequency and logarithmic decrement of copper single crystal at 20°K vs reactor time in kilowatt hours. The left-hand ordinate is the resonant frequency in cycles per second of the crystal, values of which are given by circles (●). The right-hand ordinate measures the logarithmic decrement (▲).

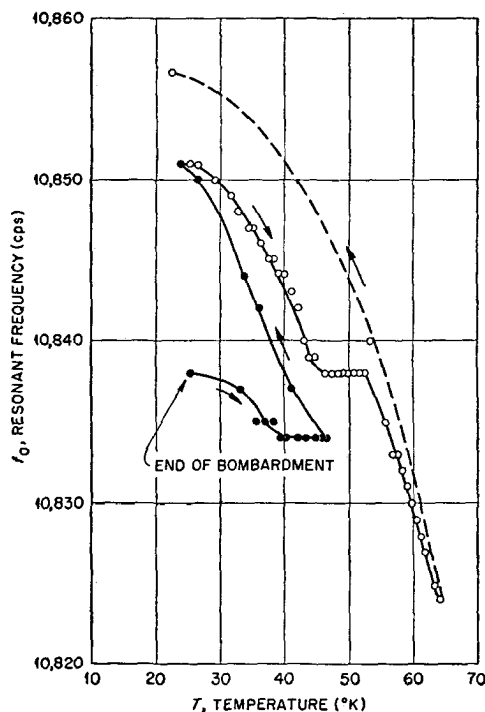


FIG. 2. "Annealing" data for copper single crystal. The left-hand ordinate measures the resonant frequency of the crystal. Filled circles (●) represent the first warmup and return to about 21°K, and the open circles (○) represent the second warmup. The abscissa is the temperature in degrees Kelvin.

to 21°K, the highest value of the resonant frequency and lowest value of internal friction were reached. The values of resonant frequency and internal friction after these warm-up runs are shown in relation to the bombardment curves in Fig. 1, and are connected by a wavy line.

Following the last warm-up curves, the reactor was again turned on and the crystal was bombarded for a week at approximately 20°K. Measurements of both the modulus and internal friction were made throughout this bombardment. No significant changes in either the resonant frequency or decrement were observed during this time. Thus it can be concluded that the fractional change in the modulus during this irradiation was less than the fractional change in the crystal length due to the irradiation.

It is interesting to note that a warmup after a week's bombardment gave no indication of a knee, as indicated in Fig. 2 for the first two warmups. Thus, it seems certain that the "annealing" process previously mentioned, as it affects the dislocation modulus and internal friction measurements, was fairly well completed after the second warmup in Fig. 2.

The data presented may constitute evidence for interstitialcy motion<sup>3-5</sup> at a temperature of about 21°K in which the interstitialcy reaches a dislocation as a trapping site. If dislocation pinning is assumed, as seems quite well established,<sup>2</sup> the change in modulus follows the equation

$$\frac{1}{E(t)} = \frac{1}{E_e} \frac{C}{(1+\gamma t)^2} \quad (2)$$

in which  $E(t)$  is the modulus measured as a function of time (or irradiation),  $E_e$  is the final value of the modulus,  $C$  and  $\gamma$  are constants, and  $t$  is the time. From the experimentally determined constants in this equation and from previously determined dislocation densities, it can be concluded that the range of damage propagation is about 150 atom spacings. Since this range is some 15 or more times larger than the region over which direct neutron

damage can be expected to extend from neutron energy considerations, and since all known defects capable of pinning dislocations are immobile at these temperatures, it is clear that another mechanism of damage propagation is necessary in order to interpret the results. Suggestions for such means of damage propagation are (1) interstitial atoms moving by the interstitialcy<sup>4</sup> mechanism, or (2) interstitial atoms in the form of crowdions<sup>5</sup> moving along a close-packed line of atoms.

<sup>1</sup> Coltman, Blewitt, and Noggle, submitted to *Rev. Sci. Instr.* **28**, 375 (1957).

<sup>2</sup> D. O. Thompson and D. K. Holmes, *J. Appl. Phys.* **27**, 713 (1956).

<sup>3</sup> H. B. Huntington, *Phys. Rev.* **91**, 1092 (1953).

<sup>4</sup> G. H. Kinchin and R. S. Pease, *J. Nuclear Energy* **1**, 200 (1955).

<sup>5</sup> W. M. Lomer and A. H. Cottrell, *Phil. Mag.* **46**, 711 (1955).

## Dislocations Observed in a Silver Chloride Crystal\*

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(Received February 26, 1957)

REPETITIVE microsecond light flashes with simultaneous voltage pulses have been applied to large crystals of silver halide grown from the melt as well as to emulsion grains.<sup>1,2</sup> Examination of the exposed large crystals with a light microscope has shown that single dislocations within the crystal are delineated by the photolytic silver, as was demonstrated earlier by Hedges and Mitchell<sup>3</sup> for subsurface structure. Application of the pulsing technique has been especially advantageous for such a study because the photoelectrons are displaced from the surface into the crystal. This movement continues for the duration of the voltage pulse or until the electrons are permanently trapped, whichever is shorter. In agreement with observations on dislocations in germanium,<sup>4</sup> the dislocations in AgCl proved to be good electron traps, as shown by the fact that there was an enhanced formation of silver at those dislocations which were in the path of the electrons.

The results reported have been generally observed in silver halide crystals. The photomicrographs in Figs. 1(a) and 1(b) show dislocations in a AgCl crystal containing 1 ppm of copper grown by a modified Bridgman technique. Photoelectrons created at the surface of the crystal during the light flash were moved into the crystal by the pulsed electric field. Following a pulsed exposure through a small aperture, an internal column of photolytic silver was formed in the region where the electrons were trapped. The crystal was then cut lengthwise through this column with a razor blade, the new surface was polished, and then examined with a light microscope.

Within this silver column, Fig. 1(a) shows two dislocations, delineated by the vertical lines of silver specks, which pass through the plane of focus of the microscope. Approximately 10<sup>5</sup> such dislocations were observed per cm<sup>2</sup> by this technique. This is several orders of magnitude lower than values given in the literature. Most of these dislocations were curved and extended

for an average of less than 100 $\mu$ . It seems probable that these lines of silver specks represent edge dislocations. The horizontal row of silver specks in the same figure extends as a plane of non-uniformly spaced silver as far as can be seen into the crystal. Such a configuration is probably caused by a slip plane in the crystal.

In contrast to this nonuniform plane of silver, Fig. 1(b) shows a row of uniformly spaced silver specks delineating what is probably a small-angle grain boundary. Such a boundary, as suggested by Burgers,<sup>5</sup> is probably a series of equally spaced dislocations. Thus, each silver speck in the row may represent a dislocation, and this is actually the case since each speck is followed by another directly beneath it, as far as can be seen by focusing down into the crystal. These specks may correspond to the etch pits reported on the surface of other materials.<sup>6</sup> The spacing between silver specks is 2 $\mu$  so that, from  $D=d/\theta$ , one obtains a rotation  $\theta$  about an axis in the boundary of 41 seconds of arc if a simple case of crystal orientation is taken where  $d$  is one-half the face diagonal of the AgCl unit cell.

The author is indebted to F. Moser, N. Nail, and W. C. York for preparing the crystal. The author is grateful to F. A. Hamm and C. R. Berry for helpful discussion.

\* Communication No. 1889 from the Kodak Research Laboratories.

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<sup>2</sup> J. H. Webb, *J. Appl. Phys.* **26**, 1309 (1955).

<sup>3</sup> J. M. Hedges and J. W. Mitchell, *Phil. Mag.* (7) **44**, 357 (1953).

<sup>4</sup> Pearson, Read, and Morin, *Phys. Rev.* **93**, 666 (1954).

<sup>5</sup> J. M. Burgers, *Proc. Phys. Soc. (London)* **52**, 23 (1940).

<sup>6</sup> Vogel, Pfann, Corey, and Thomas, *Phys. Rev.* **90**, 489 (1953).

## Technique for Photographing Falling Objects

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(Received February 15, 1957)

IN a recent article<sup>1</sup> Magarvey and Taylor described an experimental arrangement for photographing falling water drops. At our laboratory, while making measurements of the radar cross sections of water drops at millimeter wavelengths, we have used a different and more versatile technique for photographing falling water drops. Equipment was arranged so that each falling drop passed through a microwave radar beam, and the power backscattered to the radar receiver was used to operate a high-speed electronic flash system. The room in which the equipment was located was darkened before photographs were taken; the desired photograph was obtained with a camera whose shutter was open while the drop was falling, so that the exposure time was determined by the flash duration. The equipment arrangement is shown in Fig. 1.

The water drops to be photographed were released slowly from an eye dropper attached to a water reservoir at ceiling height. Drops as large as six or seven millimeters in diameter were produced with glass or wettable plastic tubes having an orifice diameter of about  $\frac{1}{8}$  inch, while smaller drops were formed with very small bore nozzles made from a nonwetting plastic such as Teflon.

Lower-power, c.w. radar systems, operated at various microwave frequencies (10 to 50 kmc), gave backscattered signals large enough for satisfactory triggering of the flash tube equipment. In each case a standard klystron provided sufficient power; horn antennas of about 20 db gain were used for transmission and reception; and conventional crystals and crystal mounts were used as detectors. (A more complete description of the synchronous detection system used has been given by Brodwin, Johnson, and Waters.<sup>2</sup>) The distance from the horns to the path of fall of the drops was usually in the vicinity of one foot.

Since the frequency of the doppler signal detected is proportional to the magnitude of the radial component of velocity between the target (water drops) and the radar set, the horn antennas were normally pointed upward at an angle of about 15°

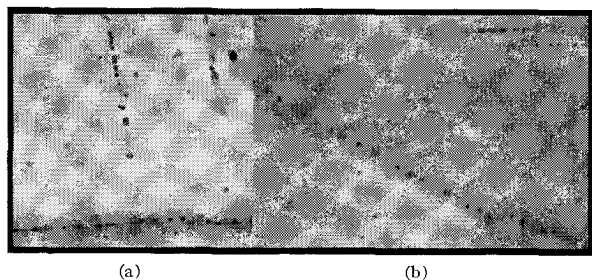


FIG. 1. (a) Edge dislocations (vertical lines) and a probable slip plane (horizontal line) delineated by photolytic silver particles in a AgCl crystal. (b) A small-angle grain boundary revealed by the uniformly spaced particles of photolytic silver.