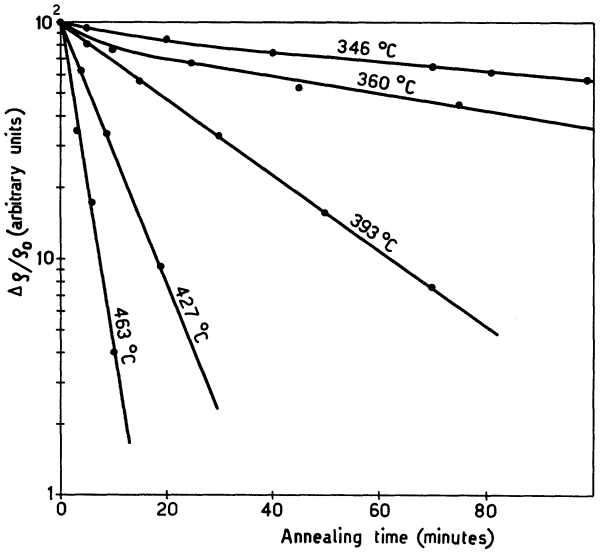

FIG. 2. Typical isothermal annealing curves. 0.04-mm wires quenched at about 930°K.

migration energy of vacancies. Recently Damask et al.[4] calculated the activation energy for migration of vacancies at 1.3 ev in copper.

The experimentally known value of the activation energy for self-diffusion in copper (2.05 ± 0.15 ev),[5] if combined with the above value of the energy for vacancy production, suggests that the migration energy of a vacancy must be somewhat larger than 1 ev, in agreement with the result by Granato et al.[6] This fact does not conflict with present annealing data.

A more accurate determination of the activation energy for migration of vacancies could have been obtained if faster quenchings were feasible: many attempts have been made to find out a suitable quenching agent, but they were unsuccessful, poor reproducibility having been achieved. Thinner copper wires might possibly be used with the purpose of getting shorter cooling times without loss of accuracy.

[1]99.999% pure, supplied by Johnson, Matthey, and Company, London.

[2]P. Jongenburger, Appl. Sci. Research B3, 237 (1953).

[3]F. Abelès, J. phys. radium 16, 345 (1955).

[4]Damask, Dienes, and Weizer, Phys. Rev. (to be published).

[5]Kuper, Letaw, Slifkin, Sonder, and Tomizuka, Phys. Rev. 96, 1224 (1954).

[6]Granato, Hikata, and Lücke, Phys. Rev. 108, 1344 (1957).

THERMAL AND RADIATION ANNEALING OF Ge*

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It is found that about 50% of the defects produced by 1.10-Mev electron irradiation of Ge at temperatures near 10°K can be annealed either by heating to 80°K or by irradiating with electrons of energy less than the threshold for damage.

Nearly degenerate single crystals of n-type Ge ($n_0 = 7\times10^{17}/\text{cm}^3$) were used in this investigation. Samples, 60 to 80μ thick, were mounted in a cryostat which permitted measurement of Hall coefficient, R, and conductivity, σ, as a function of irradiation. The sample temperature was maintained by heat exchange through low pressure He gas to a liquid He reservoir. Temperature was measured by means of two calibrated carbon resistor thermometers, one soldered to each end of the sample. The resistors were shielded from the electron beam. The rise in sample temperature during irradiation was less than 6°. All measurements of R and σ were made at 4.2°K.

Both σ and carrier concentration, n, decreased almost linearly under 1.10-Mev irradiation. The change in mobility, $\Delta(R\sigma)$, accounted for about 60% of the change in σ. The rate of removal of carriers was 2 (carriers/cm^3) per (electron/cm^2). This is about twice the value obtained for nondegenerate samples at 78°K.

Figure 1 illustrates the thermal recovery of σ after 1.10-Mev irradiation. Each point represents the value at 4.2°K after 7.5 minutes at the temperature of anneal. Two recovery regions were observed. The first, near 30°K, was well defined on all anneals. The second was more or less distinct depending on the sample and its history. No further recovery was observed between 80° and 130°K. The recovery in Fig. 1 amounts to 50% of the change under irradiation. If a first-order recovery process is assumed, the 30°K recovery occurs with an activation energy of 0.04 ev. Values for the higher temperature recovery lie between 0.06 and 0.09 ev.

These results are in substantial agreement with those of Gobeli,[1] who observed thermal recovery in both n- and p-type Ge after irradiation near 4.2°K with 3.7-Mev α-particles. Cleland

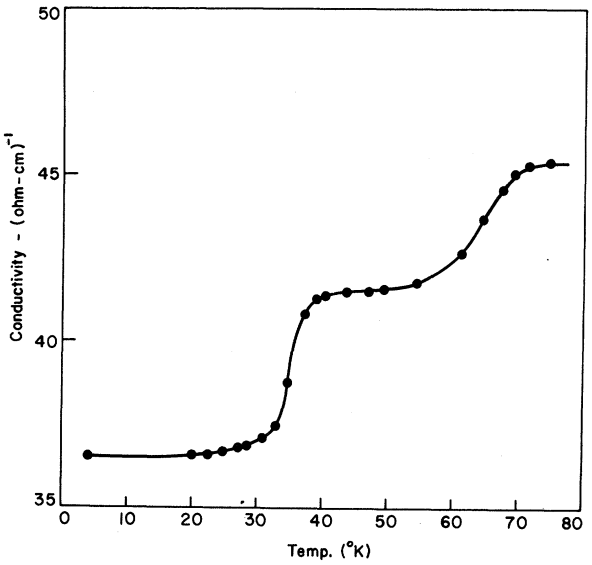

FIG. 1. Isochronal thermal recovery of conductivity in n-type Ge, after irradiation by 4×10^{16} electrons/cm^2, at 1.10 Mev.

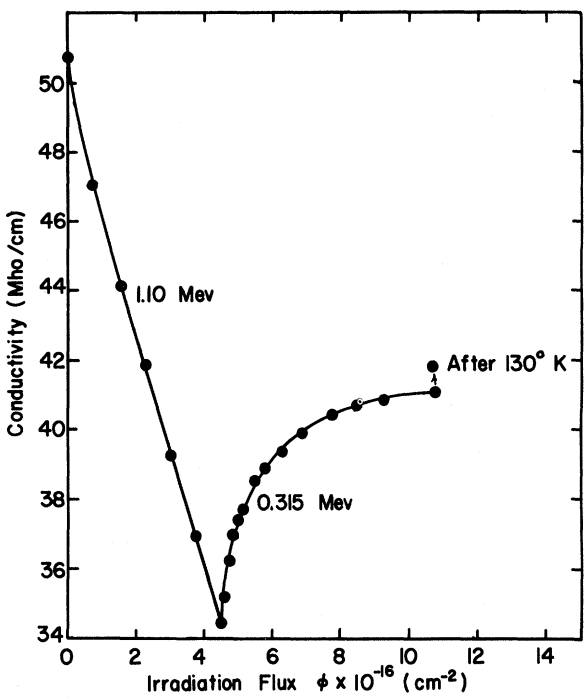

FIG. 2. Conductivity changes in n-type Ge with 1.10-Mev electron irradiation followed by 0.315-Mev irradiation.

and Crawford[2] report that they observed no recovery below 80°K for neutron-irradiated Ge. However, as they point out, their measurements were made in the presence of a strong γ-ray field. Either minority-carrier trapping or radiation-induced recovery of the defects, due to the γ-flux, may possibly account for the different results. In our experiments there was no evidence of minority-carrier trapping.

Figure 2 shows the effect of irradiation at 1.10 Mev followed by 0.315 Mev. The 1.10-Mev irradiation produced an almost linear decrease of σ, indicating little if any radiation-induced recovery. However, subsequent irradiation at 0.315 Mev, which is below the threshold for bulk damage in Ge, produced rapid recovery of σ. A similar recovery occurred for n and $R\sigma$. The recovery of n can be described by an equation of the form

$$dn/d\phi = S(n_a - n),$$

where n_a is the asymptotic carrier concentration and S is the cross section for the process. This fit yields a value of $S \simeq 10^{-16}$ cm^2 for 0.315 Mev.

The slight curvature in 1.10-Mev irradiations and rapid recovery under 0.315-Mev irradiation suggests that S decreases with increasing energy. This was verified by using 0.60-Mev electrons to produce radiation annealing after a 1.10-Mev irradiation. Although 0.60 Mev is well above the threshold for damage, analysis indicates that S for this energy is about 5×10^{-17} cm^2.

Thermal recovery and radiation-induced recovery are equivalent in that each suppresses the other and each produces about the same amount of recovery after equal irradiation at 1.10 Mev. Thermal treatment after 0.315-Mev irradiation produced only the small additional recovery indicated in Fig. 2. Irradiation by 0.315-Mev electrons after a thermal anneal at 130°K produced no measurable changes in R or σ. We regard this behavior as strong evidence that the thermal anneal resulted in the disappearance of centers introduced by 1.10-Mev irradiation. If the recovery were due to release of carriers from traps, without destruction of the trapping centers, we would expect to observe a decrease in σ and n, due to retrapping of carriers on these centers, under subsequent 0.315-Mev irradiation.

We conclude that a significant fraction of the defects produced in Ge at temperatures below 10°K, are unstable. These defects are most probably close vacancy-interstitial pairs. The very large value and the inverted energy dependence of the cross section suggest that ionization

may play a role in the radiation-induced recovery.

*This research is supported by the U. S. Atomic Energy Commission.

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[2]J. W. Cleland and J. H. Crawford, Jr., J. Appl. Phys. 29, 149 (1958).

X-RAY MEASUREMENT OF THE DISTRIBUTION OF ELECTRONS IN IRON AND COPPER

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Batterman[1] has recently reported some x-ray measurements on powdered Fe and Cu in which he concludes that the number of $3d$ electrons in Fe is 6.2 ± 0.4 measured relative to copper (assumed to have ten $3d$ electrons) and is 6.9 ± 1.0 relative to a NaCl standard. Simultaneously with reporting this he has made available to us samples of Fe and Cu powder which he used in making these determinations and we are grateful for his consideration.

We have repeated his measurements and made several of our own and while we find no major differences in the raw data, our analysis of the data fixes the number of $3d$ electrons in Fe at approximately 2.5 ± 1.5. The differences in the analyses undoubtedly arise from factors such as absorption coefficients, Hönl corrections, Debye-Waller factors, extinction corrections, and packing effects which cannot all be evaluated until Batterman reports these in detail. However, some of these differences can be discussed.

Firstly, Batterman has used tabulated Hönl corrections whereas we have measured these and find them to be -1.9 for Fe and -1.84 for Cu (Fe $K\alpha$). These have been measured by comparing the intensity ratio of the Fe 211 and Cu 311 peaks to an Al 311 peak at both Mo $K\alpha$ and Fe $K\alpha$. In this comparison the Debye-Waller factors, preferred orientation, and any uncertainty in the scattering factors approximately cancel out and the only major difference in the ratios at the two wavelengths is the Hönl correction at Fe $K\alpha$ (the correction is small at Mo $K\alpha$). Secondly, we have found large packing density differences in the integrated intensities of Fe between compressed and loose-packed samples (~25%) and believe this to be the case for NaCl since its linear absorption coefficient is not much smaller than that of Fe. Unfortunately the onset of preferred orientation in compressed NaCl prevents a direct measurement, but an indication that such an effect is present can be obtained by comparing the total integrated intensities of all the peaks in a compressed and loose-packed sample. If only preferred orientation sets in on compression, then intensity lost from some peaks will be gained by others. We have done this and find a 45% increase in the compressed sample. While some peaks are unchanged on compression, this is no doubt due to preferred orientation and packing effects cancelling each other. The precise reason for this effect is not understood as we have also observed this effect with more penetrating radiation (Mo $K\alpha$) on samples of V, Fe, and α brass. We believe that this effect may be related to the packing density since it is independent of angle. Hence the use of loose-packed NaCl to standardize compressed Fe and Cu samples will be in error. Thirdly, we find at least 12% extinction in Batterman's Cu sample as determined from the 111/222 ratio (this is free of preferred orientation). Of course this depends on the Debye temperature used (we used $\theta = 320°$),[2] and on precisely how one draws the background since the 222 peak is broad. In order to circumvent these uncertainties, we have standardized his Cu 111 peak against the 311 peak of compressed 325-mesh Al filings which were free of extinction on compression and exhibited little preferred orientation. We found his Cu 111 peak to be $(12 \pm 4)\%$ low which we believe supports our contention of extinction.

If we combine the 12% extinction correction with our measured Hönl correction, then Batterman's measured Fe 110 scattering factor is reduced from 18.9 (relative to copper) to 17.1 or, in terms of $3d$ electrons, from 6.2 to 2.8. (As a result of the changed $3d$ electron configuration a correction of ~0.5 $3d$ electron has been made due to the argon core contraction.[3])

As an independent check, we have measured the Fe 110 peak relative to the above-mentioned Al standard and obtained $f = 17.0 \pm 0.9$ with Fe $K\alpha$ and 17.0 ± 0.9 with Mo $K\alpha$. These measurements correspond to 2.5 ± 1.5 $3d$ electrons.

While the numbers we quote result from a straightforward analysis of the data, we believe that powder measurements can be taken seriously