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FOR PERIOD ENDING AUGUST 31, 1962

OAK RIDGE NATIONAL LABORATORY
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UNION CARBIDE CORPORATION
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U.S. ATOMIC ENERGY COMMISSION

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For Period Ending August 31, 1962

D. S. Billington, Director
J. H. Crawford, Jr., Associate Director

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Abstracts of published papers: *Phys. Rev.* 124, 1731 (1961); *Phys. Rev.* 126, 1342 (1962). Extensive measurements on irradiated germanium indicate that previous analyses of the recombination process are incorrect. Observations of the annealing behavior of radiation-induced recombination centers have been made. A model which explains the recombination and annealing data is presented.

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17.4 RESEARCH MATERIALS INFORMATION CENTER

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This report very briefly describes the purpose and operation of the Solid State Division's Research Materials Information Center, which will provide information on the methods of production, availability, need for, and characterization of high-purity solid-state research materials to both producers and users. Details of system automation and analysis for coding are not included.

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It appears that purification of Bi_2Se_3 can be considerably enhanced by regulation of the constituent-element vapor pressure over the growing crystal. A modified Teal-Little crystal-growing furnace has been constructed for this purpose.

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J. M. Williams, M. S. Wechsler, and H. M. Otte 137

Abstract of paper presented by H. M. Otte at International Conference on Crystal Lattice Defects, Kyoto, Japan, Sept. 7-12, 1962. The effect of room-temperature deformation and subsequent isochronal annealing on various physical properties of Cu-6.7 at. % Si and Cu-6.7 at. % Si-1.3 at. % Mn is being studied. The results are interpreted in terms of the redistribution of solute stimulated by the deformation-produced defects.

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W. E. Brundage, W. W. Davis, N. E. Hinkle, O. Sisman, J. C. Zukas, and A. L. Johnson 138

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PART VI. REACTOR MATERIALS

20. FUEL MATERIALS

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Irradiation in a reactor causes zircon ceramic to resemble metamict zircon in the changes undergone on heating. Heating produces partial annealing at relatively low temperature, decomposition at higher temperature, and re-formation of zircon at still higher temperature. Not all of these processes may occur, depending on the irradiation dose.

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Part I. Theory

D. K. Holmes

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1. Studies of Penetration of Moving Atoms in Solids and Interatomic Potentials

1.1 STOPPING POWER AS A BASIS FOR HARD-SPHERE APPROXIMATION

O. S. Oen M. T. Robinson
D. K. Holmes

The "hard-sphere approximation" has had widespread use with much success in radiation-damage theory. The main advantage of this approximation, from a mathematical viewpoint, is that it allows a simplified analytical approach to extremely complex problems. A disadvantage is the difficulty in evaluating the errors associated with it. For focusing collisions the hard-sphere approximation has been found to be very good.¹ On the other hand, its use in range calculations accounts for the general features of the more exact calculations² but gives results which are 10 to 80% too high (Bohr-potential calculations). The reason for this overestimate is that the stopping power of the Bohr potential is 10% (at low energy) to 80% (at high energy) greater than that of the approximating hard-sphere potential. This increased stopping power tends to shorten the calculated ranges. These results suggested that a new criterion for the hard-sphere radius might be more appropriate for range calculations.

The hard-sphere radius is usually defined as the distance of closest approach between two atoms in a head-on collision. An alternative method is to define it so that the hard-sphere stopping power matches the exact, calculated stopping power. This is done in Eq. (1), where, for simplicity, the

masses of the primary atom and lattice atoms are assumed to be equal:

$$\frac{n\pi R_{s.p.}^2 E}{2} = n \int_0^E T K(T, E) dT, \quad (1)$$

where the left-hand expression is the hard-sphere stopping power, and the right-hand expression is the calculated stopping power for the potential being approximated. In Eq. (1), n is the density of atoms, E is the primary energy, $K(T, E)$ is the differential cross section for transferring an energy T to a lattice atom by a primary of energy E and $R_{s.p.}$ is the hard-sphere radius chosen to make the two expressions equal. In order to compare the ranges obtained by the two methods of choosing the hard-sphere radius to approximate the Bohr potential, the results of Figs. 1.1 and 1.2 have been calculated using the hard-sphere analytical range formalism of Holmes and Leibfried.³ Figure 1.1 shows that the average penetration depth calculated with a radius chosen by matching the stopping powers gives very good agreement with the Monte Carlo results. This method also gives agreement to better than 5% of the Monte Carlo calculations for the total path and vector range. However, as seen from Fig. 1.2, the perpendicular spread is represented somewhat better by using a radius defined by the distance of closest approach.

It is concluded that, for theoretical estimates of ranges, this new method of selecting the hard-core radius is superior to the usual method, at least for simulating the Bohr potential. On the other hand, to estimate the lateral spread of, for example, a damaged region initiated by a primary knock-on, the usual method of picking a hard-sphere radius seems to be better.

¹C. Lehmann and G. Leibfried, *Z. Physik* **162**, 203 (1961).

²M. T. Robinson, D. K. Holmes, and O. S. Oen, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 3.

³D. K. Holmes and G. Leibfried, *J. Appl. Phys.* **31**, 1046 (1960).

Fig. 1.1. Comparison of the Penetration Depth as a Function of Primary Energy for Two Choices of the Hard-Sphere Radius and the Exact Calculation.

Fig. 1.2. Comparison of the "Spread" as a Function of Primary Energy for Two Choices of Hard-Sphere Radius and the Exact Calculation.

Frequently, the range of an energetic atom in a solid is calculated by integrating the reciprocal of the atomic stopping power.^{4,5} (In principle, energy losses by electron excitation and ionization can be included also. For simplicity, they are not included in this discussion.) As is well known, this method should be quite accurate, provided the atomic weight of the stopping material is much less than that of the projectile atom. This requirement is seldom met in radiation damage where interest usually lies in the range of an atom having the same atomic weight as that of the stopping material. However, even in this case, the range found by integrating the reciprocal of the stopping power is expected to give the average total path

⁴K. O. Nielsen, p 68 in *Electromagnetically Enriched Isotopes and Mass Spectrometry*, Academic Press, New York, 1956.

⁵J. Linhard and M. Scharff, *Phys. Rev.* 124, 128 (1961).

length traversed by the projectile. It is interesting to compare this "stopping power range" with the total path found by the discrete-energy-loss method for atoms of equal mass. As an example, the hard-sphere approximation to the Bohr potential will be discussed. The hard-sphere stopping power is

$$-\frac{\Delta E}{\Delta L} = \frac{\pi \pi R^2 E}{2}, \quad (2)$$

where R is now defined by

$$E = \frac{E_B}{R/a} e^{-R/a}. \quad (3)$$

Here E_B is the Bohr energy and a is the screening length. The "stopping power range," L^* , is found to be

$$L^* = 2\lambda,$$

whereas the discrete-energy-loss method of Holmes and Leibfried gives a total path, L , equal to

$$L = \lambda(3/2 + R_i/a).$$

In the above formulas, $\lambda = 1/\pi \pi R_i^2$, where R_i is the hard-sphere radius at the initial energy of the primary. It is noted that the "stopping power range" may be greater or less than that found by the discrete-energy-loss method, depending on whether R_i/a is less or greater than $1/2$. This surprising result can be elucidated by examining the stopping power [Eq. (1)] shown as the solid line in Fig. 1.3.

When the range is determined by integrating the reciprocal of the stopping power, the particle is assumed to lose energy in a continuous manner; thus it passes through all points of the $-\Delta E/\Delta L$ curve. On the other hand, in the method of discrete energy loss, the particle loses energy in arbitrary discrete amounts and therefore the "effective stopping power" is that shown by the dotted line (Fig. 1.3). It is seen that the stopping power may be greater or less than the "effective stopping power," which affects the calculated "ranges" in a corresponding manner.

The "stopping power range" using the exact, calculated stopping power of the Bohr potential has been evaluated and compared with the results of the Monte Carlo calculations. The results found were similar to those discussed above and could be interpreted in an analogous manner. For

Fig. 1.3. Schematic Diagram of Hard-Sphere Stopping Power (Solid Line). The dotted line is the effective stopping power of a typical atom slowing down by discrete energy losses.

the Nielsen C/r^2 potential, the stopping power is a constant, independent of energy; therefore, it would be predicted that the two ranges would be equal, which, indeed, turns out to be true.

In conclusion, this method of examining the stopping power curve seems very useful in obtaining an estimate of the accuracy of the "range" found by integrating the reciprocal of the stopping power.

1.2 MACHINE STUDIES OF THE SLOWING DOWN OF ENERGETIC ATOMS IN SOLIDS

M. T. Robinson O. S. Oen

Several papers⁶⁻¹⁰ have been prepared describing various results from a series of machine calculations¹¹ of the slowing down of energetic

⁶M. T. Robinson, D. K. Holmes, and O. S. Oen, *Proceedings of the International Colloquium on Ion Bombardment, Paris, 1961* (to be published by Centre National de la Recherche Scientifique).

⁷O. S. Oen, M. T. Robinson, and D. K. Holmes, *Bull. Am. Phys. Soc.* 7(2), 171 (1962).

⁸M. T. Robinson, O. S. Oen, and D. K. Holmes, *Bull. Am. Phys. Soc.* 7(1), 171 (1962).

⁹D. K. Holmes, to be published in *Proceedings of the International Atomic Energy Agency Symposium on Radiation Damage in Solids and Reactor Materials, Venice, 1962*.

¹⁰O. S. Oen, D. K. Holmes, and M. T. Robinson, submitted to the *Journal of Applied Physics*.

¹¹M. T. Robinson, D. K. Holmes, and O. S. Oen, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 3.

atoms in solids. The calculations are based on the assumption of purely elastic scattering encounters between atoms, so that the energy range of interest is below 100 kev. The histories of individual moving atoms are followed, using Monte Carlo techniques, as the atoms slow down in moving through the crystal. The potential of interaction on which most of the effort to date has been expended is the exponentially screened Coulomb or Bohr potential. In the Monte Carlo studies, the scattering angles are accurately calculated at each collision, using the classical scattering formulas. The results have been compared with approximate calculations in which the screened Coulomb potential has been replaced by a fitted hard-sphere potential or by a fitted inverse r -squared potential; neither of these gives completely acceptable results. The ranges of the atoms have been compared with experimental results, and it has been shown that the simple Bohr potential, using Bohr's screening radius, is inadequate in the sense that a stronger Z dependence than that assumed by Bohr is needed to give adequate correspondence with experiment. With one exception,⁸ these papers dealt with calculations performed with an IBM 7090 program designated as RANGE I, in which the atoms of the solid were assumed to be randomly located in space. The correct density of the solid was maintained by choosing a constant "free flight" distance between collisions. A new program, RANGE II, has been written in which the atoms of the solid are correctly located on lattice sites, obviating the need to use the rather unsatisfactory concept of free flight. The opportunity was also taken to improve the treatment of the equations of motion of the particles involved in a collision and to elaborate the analysis of the results of the computations. In addition to distributions and central measures of various range quantities, the new program produces distributions of the final primary energy and of the final primary direction.

RANGE II permits the study of solids having any structure conveniently describable on orthorhombic coordinates (this includes hexagonal crystals, for example, but excludes many monoclinic and triclinic ones) and having as many as nine crystallographically distinct atoms (these may or may not be chemically different). A simple model of thermal vibrations is included, each kind of atom being permitted to execute simple harmonic motions about its lattice site with an

appropriate rms amplitude. Other new features of the program include the possibilities of following the primary for a specified number of collisions (rather than through a specified energy range) and of truncating any histories exceeding a preset number of collisions. Various sorts of detailed output of individual histories are also permitted. The new program requires 50 to 150% more computer time than did RANGE I, the increase depending on which combination of several options is chosen.

Most of the results obtained with the RANGE II program refer to the following "standard problem": 10-kev copper atoms are started isotropically from a lattice site in an infinite crystal of copper. They are permitted to lose energy in two-body interactions with the atoms of the crystal until their energies fall below 25 ev. Unless otherwise stated, this standard problem will be the source of the results cited below.

The most striking result of the calculations is that a small fraction of the primary particles ($\sim 1\%$, depending on the potential chosen and the thermal vibration amplitude) move exceedingly large distances with only very small losses in energy. Such events were anticipated for hard-core calculations, and a means was provided in the program for rejection of those primaries which traveled (at any energy) more than a preset distance. In the standard problem this distance was chosen as (approximately)

$$a_0^3/[R(10 \text{ kev})]^2 + 25\sqrt{6} a_0 \approx 880 \text{ A},$$

where a_0 is the lattice constant of copper, $R(10 \text{ kev})$ is the hard-core radius appropriate to the initial energy, and the second term represents the average distance for 100 collisions to occur. [The average distance which a primary moves between collisions in the fcc metals is approximately $\frac{1}{2}$ the unit translation in a $\langle 112 \rangle$ direction, $(\frac{3}{2})^{1/2} a_0$. This result is true of the calculations made for Al, Cu, Ag, and Au with the RANGE I program as well as RANGE II.] In the standard program, 0.5% of the primaries were rejected on this score, the others all having ranges of ordinary magnitude. The surprising result was the presence of a similar group of particles in calculations made with the Bohr and with the Born-Mayer potentials. The occurrence of these "wild" histories in the latter cases required numerous changes in the program to avoid excessive consumption of machine

time and has temporarily interrupted the computational program because of their disastrous effects on many of the problems of interest.

A study of these penetrating events, which have become known as "channel" or "tunnel" events, reveals that the majority of them are moving very close to the $\langle 110 \rangle$ directions of the lattice. A portion of a typical trajectory is shown in Fig. 1.4. The particle spirals leisurely down the channel in the $[10\bar{1}]$ direction, its motion very largely restricted to a single plane. The stereographic triangle of Fig. 1.5 shows the directions of the 20 particles (out of 1009) in the standard

problem which made over 250 collisions (the mean number of collisions in slowing down to 25 ev was 57.6). Nine of these particles made over 1000 collisions; their directions after the 800th collision are also shown in the figure. It is evident that a sort of focusing is operative and that the $\langle 110 \rangle$ direction is preferred. That this is not the only direction in which "channeling" is significant, however, is shown by a run in which 10-kev copper primaries were normally incident on a (001) surface of copper. In this case, 75% of the primaries made more than 250 collisions; in fact, only 300 out of 1188 particles reached 25 ev before

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AT THE ORIGIN OF THE COORDINATE SYSTEM,
THE PROJECTILE ENERGY IS 387 ev
COLLISION NUMBERS ARE INDICATED ALONG
THE TRAJECTORY. THE TOTAL NUMBER OF
COLLISIONS IN THIS HISTORY WAS 10567.

PROJECTION ONTO
THE $(10\bar{1})$ PLANE
SCALES IN ANGSTROMS
CIRCLES SHOW
LOCATIONS OF ATOMS
IN THE PROJECTION

Fig. 1.4. Portion of a "Channel" Event. A 10-kev copper primary started from a lattice site. The Bohr potential ($k = 1$) was used.

Fig. 1.5. Directions of the Longest Trajectories of 10-keV Copper Primaries Slowing Down in Copper According to the Bohr Potential ($k = 1$).

the computer run was arbitrarily terminated. All the very long trajectories were moving very nearly in the [001] direction, and only small energy losses had occurred.

If the "channel" events are ignored, comparisons of the lattice and random calculations can be made. These comparisons are given in Figs. 1.6 and 1.7 and in Table 1.1. With the use of the hard-core approximation (Fig. 1.6), the two solid models give results in quite close agreement. However, when the Bohr potential is employed (Fig. 1.7), the distribution of the vector range is seen to be significantly more skewed in the lattice model than in the random case. The shape of the distribution in the lattice case is more nearly like the experimental observations of Davies¹² than is that in the random case. Whether the channel events are to be identified with the exponential tail observed by Davies is still a very open question.

¹²J. A. Davies *et al.*, *Can. J. Chem.* 38, 1535 (1960).

Fig. 1.6. Distribution of the Vector Range, 10-keV Copper Atoms Slowing Down to 25 eV in Copper. A comparison of the lattice and random models for hard-core interactions.

Fig. 1.7. Distribution of the Vector Range, 10-kev Copper Atoms Slowing Down to 25 ev in Copper. A comparison of the Lattice and random models for the Bohr potential.

The effects of thermal vibration on the vector range and the occurrence of channel events are shown in Table 1.2. The variations in the range quantities are probably due to statistical fluctuations, but the number of channel events decreases to zero if a sufficiently large "temperature" is employed.

The channel events change in identity and number when superficially trivial changes are made in the potential, affecting only the potential at large separations. This fact suggests that much of what is seen in the calculations is due to unsatisfactory treatment of the scattering problem at large distances. For this reason, present efforts are being directed toward improvements in the computational procedure so that more satisfactory potentials may be used. The approach currently being investigated involves the developing of approximations to the scattering integrals for the interaction between two particles in the form

$$T/T_{\max} = \frac{1 + \alpha_1 b^2 + \alpha_2 b^4 + \dots}{1 + \beta_1 b^2 + \beta_2 b^4 + \dots}, \quad (1)$$

where the α_i and β_i are functions of energy and b is the impact parameter. The well-known hard-core approximation may be represented by

$$\left. \begin{aligned} \beta_1 = \beta_2 = \dots = \alpha_2 = \alpha_3 = \dots = 0 \\ \alpha_1 = -1/R^2 \end{aligned} \right\}, \quad (2)$$

where R is the hard-core radius. The truncated Coulomb approximation¹³ may be represented by

$$\left. \begin{aligned} \alpha_2 = \alpha_3 = \dots = \beta_2 = \beta_3 = \dots = 0 \\ \beta_1 = 4E^*(B + E^*)/A^2 \\ \alpha_1 = -B^2/A^2 \end{aligned} \right\}, \quad (3)$$

¹³G. Leibfried and O. S. Oen, *J. Appl. Phys.* **33**, 2257 (1962).

Table 1.1. Comparison of Solid Models and Interatomic Potentials
(10-kev copper atoms slowing down to 25 ev in copper)

	Vector Range (A)			Median	RMS
	Median	Mean	RMS	Mean	Mean
Bohr Potential ($k = 1$)					
Random	97	104	116	0.93	1.12
Lattice	88	108	149	0.81	1.38
Hard-Core Approximation					
Random	104	124	146	0.83	1.18
Lattice	104	125	154	0.83	1.23
Number of Collisions					
	Median	Mean	RMS	Total Path Collision (A)	
Bohr Potential ($k = 1$)					
Random	59	61.9	67.8	2.21	
Lattice	50	57.6	74.2	2.27	
Hard-Core Approximation					
Random ^a		7		22	
Lattice	6	7.18	7.60	21.2	
Theory (random)		6.99			

^aEstimated. The manner in which collisions were counted was not strictly comparable in the two programs when the hard-core approximation was employed.

Table 1.2. Effect of Thermal Vibrations on Vector Range
10-kev copper atoms slowing down to 25 ev in copper. Bohr potential ($k = 1$)

RMS Thermal Vibration Amplitude (A)	Vector Range (A)			Median	RMS	Channel Events (%)
	Median	Mean	RMS	Mean	Mean	
0	88	108	149	0.81	1.38	0.9
0.025	84	107	150	0.79	1.40	0.4
0.050 ^a	88	113	152	0.78	1.35	0.1
0.075	88	115	160	0.77	1.39	0.3
0.100	88	109	143	0.81	1.31	0.0
0.200 ^b	86	108	133	0.80	1.23	0.0
Random model	97	104	116	0.93	1.12	

^aApproximately 0°K for copper.

^bApproximately the melting point of copper.

where the potential is

$$V_{TC}(r) = \frac{A}{r} - B \quad 0 \leq r \leq A/B,$$

$$= 0 \quad A/B \leq r \leq \infty,$$

and E^* is the reduced energy of the primary. Since the parameters, Eq. (3), give a very reasonable account of $T/T_{\max}$ for the Bohr potential, it is hoped that rather low-order polynomials will suffice to represent any interesting potential.

1.3 HARD-SPHERE SCATTERING APPROXIMATIONS

E. J. Lee M. T. Robinson

The Holmes-Leibfried energy-dependent hard-sphere approximation¹⁴ has been compared with the approximation of Beeler and Besco¹⁵ in which the hard-sphere radius is dependent on both the incident particle energy and the impact parameter. In the Holmes-Leibfried approximation, the center-of-mass scattering angle, θ , is given by $\cos \theta = 2(s/x)^2 - 1$, where s is the impact parameter and x is the distance of closest approach for head-on collision. In the Beeler-Besco approximation, the center-of-mass scattering angle is given by $\cos \theta = 2(s/R_0)^2 - 1$, where R_0 is the distance of closest approach for the given impact parameter, s . The two approximations and an accurate numerical solution obtained on an IBM 7090 computer are compared in Fig. 1.8 for an incident energy of 5 kev in copper using the Bohr potential (exponentially screened Coulomb). The ratio of the energy transferred to the target particle to the maximum energy transferable, $T/T_{\max} = \frac{1}{2}(1 - \cos \theta)$, is plotted as a function of impact parameter in Fig. 1.8. In the numerical solution the potential was truncated at a radius of 10.4942 screening radius units. Only a negligible error results from this truncation. The curves shown are typical and illustrate the overestimation of energy transfer

¹⁴D. K. Holmes and G. Leibfried, *J. Appl. Phys.* 31, 1050 (1960).

¹⁵J. R. Beeler, Jr., and D. G. Besco, "Knock-On Cascades and Point Defect Configurations in Binary Materials," to be published in *International Atomic Energy Agency Symposium on Radiation Damage in Solids and Reactor Materials*, Venice, May 7-11, 1962.

Fig. 1.8. Energy Transfer for Cu-Cu Collisions at 5 kev Incident Energy; Bohr Potential.

for all collisions by the Beeler-Besco approximation. The disagreement increases at higher incident energies. For the closer collisions the Holmes-Leibfried approximation also overestimates the energy transfer, although to a lesser extent. On the other hand, it underestimates the energy transfer for more distant collisions. It is difficult to compare the detailed effect of the approximations on the range and other averages of interest because of the analytical complexity.

1.4 MONTE CARLO CALCULATIONS OF THE RANGES OF ENERGETIC ATOMS IN SOLIDS¹⁶

M. T. Robinson D. K. Holmes
O. S. Oen

The slowing down of energetic atoms in solids has been investigated through the use of Monte Carlo methods on a large digital computer. Neither the hard-sphere approximation nor the inverse-square approximation affords a particularly satisfactory account of the exponentially screened

¹⁶Abstract of paper presented at the *International Colloquium on Ion Bombardment*, Bellevue, France, December 4-8, 1961 (to be published).

Coulomb potential. It is found that, for copper, silver, and gold, slowing down in their respective solids, the screening radii must be, respectively, 1.15, 1.6, and 2.3 times greater than the values proposed by Bohr, in order to explain the available experimental data. It is apparent from the results of these calculations that modifications of the interatomic potential functions, especially for heavier elements, must be sought.

1.5 RANGES OF ENERGETIC ATOMS IN SOLIDS¹⁷

O. S. Oen D. K. Holmes
M. T. Robinson

The ranges in solids of atoms having energies from 1 to 100 kev have been calculated using Monte Carlo techniques. The model assumes that the moving atom loses all its energy through binary elastic collisions with the atoms of the solid. The potential of interaction principally studied is an exponentially screened Coulomb (Bohr) potential, and the scattering angles are calculated explicitly. It is found that neither the hard-sphere approximation nor the inverse r -squared approximation to the Bohr potential is particularly good. To obtain correspondence with experimental results, the Bohr screening length must be increased as the atomic number of the interacting atoms increases.

1.6 CALCULATION OF AVERAGES FOR PRIMARY RECOIL DISTRIBUTIONS¹⁸

G. Leibfried

The slowing down of an energetic particle in a random assembly of scattering centers is discussed. Certain averages for physically interesting quantities can be calculated in a very general form, including unequal masses of scattering and slowing down particles. The averages refer to quantities

such as the total distance traveled (L) and the vector distance to the final positions (x_f) and powers thereof. Two assumptions simplify the evaluation of the general results considerably: (α) The mean free path λ is a simple power of the energy E : $\lambda(E) \sim E^\mu$; (β) the distribution $g(E; \epsilon)$ of energies ϵ after one collision with initial energy E does not change its shape with E : $Eg(E; \epsilon) = \kappa(\epsilon/E)$. A special distribution of this kind is the hard core distribution. Under assumptions α and β all calculations are reduced to the evaluation of an integral like $\int \kappa(\eta) \eta^\mu d\eta$. For equal masses and the hard core distribution, one can easily calculate averages of any power of L and obtain the distribution function of L , this being proportional to $L^{1/\mu} \exp(-L/\lambda)$. Under the assumption β alone, with arbitrary $\lambda(E)$, results are given for special distributions and equal masses (including the hard core distribution). The evaluation of the averages now requires simple integrals over λ for linear averages and double integrals for quadratic averages with known integral kernels. For unequal masses, approximations are discussed.

1.7 SIMPLE APPROXIMATION FOR CLASSICAL SCATTERING AT LARGE ANGLES¹⁹

G. Leibfried O. S. Oen

The classical scattering for repulsive potentials is treated approximately by a matching method. The matching potential is a cut-off potential which matches the value and the slope of the actual potential at the distance of closest approach. The method is thought to be successfully applicable to interatomic potentials of the screened Coulomb type. Comparison is made with machine results for an exponentially screened Coulomb potential. The differential cross section and the stopping power are compared for various energies. The agreement in the differential cross section is fair except for very low energy transfers. The stopping powers agree over a wide energy range with an error of only a few percent. Further comparison is made with another approximation given by Lehman and Shapiro.

¹⁷Abstract of paper submitted for publication in the *Journal of Applied Physics*.

¹⁸Abstract of published paper: *J. Appl. Phys.* **33**, 1933 (1962).

¹⁹Abstract of published paper: *J. Appl. Phys.* **33**, 2257 (1962).

1.8 HIGHER-ORDER AVERAGES OF PRIMARY RECOIL DISTRIBUTIONS²⁰

G. Leibfried

One problem of interest in radiation damage theory is the slowing down of an energetic primary knock-on in its own lattice. The primary starts at the origin, slows down and comes to rest at a final vector distance, $r = (x_s)$. In this paper we study averages over the distribution $w(r)$ of the final positions. The slowing down is described here by a series of subsequent and independent two-body collisions, with the scattering centers located at random rather than in a lattice arrangement. Analytical expressions for the energy-dependent linear $(\overline{x_s})$ and quadratic $(\overline{x_s x_t})$ averages over $w(r)$ have been given in previous work. The purpose of this paper is to give a more elegant method which also is easily extended to higher order averages, $(\overline{x_s x_t x_u})$, $(\overline{x_s x_t x_u x_v})$, and so on. These higher order averages improve the knowledge of the distribution, which, of course, would be the most desirable quantity to calculate.

1.9 LONG-RANGE CHANNELING EFFECTS IN IRRADIATED CRYSTALS?²¹

C. Lehmann G. Leibfried

If an energetic atom, a primary, slows down in a lattice, it has the possibility of traveling long

distances, without essential interaction, along almost force-free channels bordered by close-packed atomic chains. This effect has not been taken into account until recently, because, for the sake of simplicity, the lattice always has been replaced by a corresponding random arrangement. This "channeling" behavior has been found in machine calculations by Robinson, Holmes, and Oen,²² which take the lattice structure into account. In this paper the behavior of a primary moving along a channel bordered by the most densely packed directions is investigated analytically. Numerical results are given for a copper primary moving in a copper crystal, but the results can be easily extended to other cases. Two potentials are used: (1) an exponentially screened Coulomb potential after Bohr, used also in the machine calculations and thought to give an adequate description for relatively high energies and small interatomic distances; and (2) a purely exponential potential after Born-Mayer, better suited for relatively low energies and large atomic distances. The maximum ranges are very large; for 10 kev they are of the order of 10^3 lattice parameters for the Born-Mayer potential and up to 10^7 for the Bohr potential. Presumably, the Born-Mayer potential is a better description for these events. The investigation is confined to motion near the channel axis. Therefore, we can only obtain the range distribution near the maximum range. The probability of these events is such that they can play a role in high-energy sputtering from crystal surfaces and in radiation damage.

²⁰ Abstract of paper submitted for publication in *Zeitschrift fuer Physik*.

²¹ Abstract of paper to be submitted for publication.

²² To be published; cf. also *Bull. Am. Phys. Soc.* 7(2), 171 (1962).

2. Theory of Sputtering

2.1 A GEOMETRICAL MODEL OF MONOCRYSTAL SPUTTERING

M. T. Robinson

Experimental studies¹⁻⁵ of the sputtering of copper monocrystals by rare-gas ions in the kev range have demonstrated the dramatic dependence of the sputtering yield (the number of target atoms ejected per incident ion) on the orientation of the ion beam with respect to the crystallographic axes of the target crystal. Similar experiments^{1,2} on germanium with 1- to 5-kev Ar⁺ ions reveal only a small dependence on orientation. Some success has been had in understanding the copper data on the basis of a simple, essentially geometrical, model,¹ and studies are in progress, both to improve the calculations and to extend them to targets of other symmetries.

The model assumes, following Rol,³ that the yield is determined to a sufficient degree by the first collision of the incident ion with a lattice atom. It may be mentioned, in support of this assumption, that the range of an ion in a solid is also largely determined by the first flight, at least for not too low energies.⁶ The yield, S , is then assumed to be given by

$$S_{hkl}(E) = \alpha \tau_{hkl}(E) / \lambda_{hkl}(E), \quad (1)$$

¹A. L. Southern, W. R. Willis, and M. T. Robinson, "Sputtering Experiments with 1- to 5-kev Ar⁺ ions," to be published in the *Journal of Applied Physics* (see abstract in Part III of this report).

²A. L. Southern, M. T. Robinson, and D. R. Burrowbridge, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 49; "Ar⁺ Ion Bombardment of Metal Surfaces," this report, Part III.

³P. K. Rol, Thesis, Amsterdam, 1960.

⁴O. Almén and G. Bruce, *Nucl. Instr. Methods* **11**, 257 (1961).

⁵G. D. Magnuson, General Dynamics/Astronautics, private communication, 1962.

⁶D. K. Holmes and G. Leibfried, *J. Appl. Phys.* **31**, 1046 (1960).

where E is the (laboratory) energy of the incident ion, α is a constant depending on the masses of the ions and lattice atoms, $\lambda_{hkl}(E)$ is the mean free path of an ion incident in an $\langle hkl \rangle$ direction, and $\tau_{hkl}(E)$ describes the deposition of energy by the ion in its first collision. If Rol's original notion were followed, as was done in the first application of this theory to monocrystals,¹ we would write

$$\tau_{hkl}^{(1)}(E) = 2(\overline{T}/T_{\max})E = 2(\overline{\sin^2 \theta/2})E, \quad (2)$$

where θ is the center-of-mass scattering angle, T is the transferred energy, and the overlines represent average values. The results of the original analysis of the copper data suggested that a better alternative might be to write

$$\begin{aligned} \tau_{hkl}^{(2)}(E) &= (3/2)(\overline{\sin \theta/2})E^{1/2} \\ &= (3/2)(\overline{|p|}/|p_{\max}|)E^{1/2}, \end{aligned} \quad (3)$$

where $|p|$ represents the magnitude of the transferred momentum. Since convincing arguments with respect to a choice between these two alternatives have not been developed, both will be retained for the present. Also, for the present, consideration will be restricted to hard-core scattering of the incident ions by the lattice atoms; the appropriate hard-core radius $R(E)$ will be chosen by any desired procedure. In hard-core scattering, if the impact parameter, b , could cover the entire range $0 \leq b \leq R$, there would be obtained

$$\tau_{hkl}^{(1)}(E) = E$$

and

$$\tau_{hkl}^{(2)}(E) = E^{1/2},$$

but, as pointed out before,¹ the impact parameter range in a crystal is restricted by shadowing

effects, which differ for different directions; therefore, crystallographic variability must be retained in the expressions for $\tau_{hkl}^{(1)}$ and $\tau_{hkl}^{(2)}$.

The target crystal is imagined to consist of an array of spheres of radius $R(E)$, arranged on an appropriate lattice, and the beam of ions is taken to be incident on it in an $\langle hkl \rangle$ direction. Consideration will be restricted to the case of normally incident ions. A fraction of the ions, $P_{hkl}(E)$, will undergo collisions between the surface of the crystal and a depth t_{hkl} , the unit translation parallel to $\langle hkl \rangle$, and in so doing will travel a mean distance $\bar{X}_{hkl}$. The remaining fraction of particles, $1 - P_{hkl}(E)$, will travel indefinitely far without collision, because of the repeating structure of the crystal. This manifest difficulty may be treated in either of two ways: (1) As was done before,¹ it may be assumed that those ions not colliding in $0 \leq x \leq t_{hkl}$ have mean free paths, measured from the target surface, which are isotropic. Thus,

$$\lambda_{hkl}^{(1)}(E) = P_{hkl}(E) \bar{X}_{hkl}(E) + [1 - P_{hkl}(E)] \lambda_0(E), \quad (4)$$

where

$$\lambda_0(E) = \{n\pi[R(E)]^2\}^{-1}, \quad (5)$$

in which n is the atomic density; the energy dependences indicated in Eq. (4) are all consequent on the energy dependence of the hard-core radius. (2) The other approach, which we will term the quasi-random, assumes that those particles penetrating beyond a depth t_{hkl} may be regarded as uniformly distributed in space, thus leading to the result:

$$\lambda_{hkl}^{(2)}(E) = P_{hkl}(E) \bar{X}_{hkl}(E) + [1 - P_{hkl}(E)] [t_{hkl} + \lambda_{hkl}^{(2)}(E)]$$

or

$$\lambda_{hkl}^{(2)}(E) = \bar{X}_{hkl}(E) + [1 - P_{hkl}(E)] t_{hkl} / P_{hkl}(E). \quad (6)$$

The sputtering yield may now be evaluated by inserting either Eq. (2) or Eq. (3) and either Eq. (4) or Eq. (6) into Eq. (1). The usefulness of the resulting expression is to be assessed by deter-

mining $R(E)$ and α by comparison of the equation with experimental data.

An IBM 7090 program has been written to permit the rapid evaluation of Eqs. (2) to (6) and the associated yields for a variety of different crystals. These fall into three groups:

1. A face-centered cubic group employing four sets of positions of the space groups $F43m$. Any of the four sets of fourfold positions may be occupied completely or statistically by atoms of specified mass and radius; the atoms on the different sublattices may be different. If the a sublattice alone is filled, the structure is that of the fcc metals; the b sublattice represents the octahedral holes, and the c and d sublattices represent the tetrahedral holes. The structures of InSb or germanium may be represented by filling the a and c sites, and that of NaCl is represented by filling the a and b sites.

2. A body-centered cubic group employing three sets of positions of space group $Im\bar{3}m$. The twofold a sites are those of the bcc metals, the sixfold b sites are the (distorted) octahedral holes, and the eightfold c sites have tetrahedral symmetry if all a and b sites are occupied.

3. A hexagonal close-packed group based on three sets of positions of space group $P6_3mc$. The twofold a sites are those of the hcp metals, the twofold b sites are the octahedral holes, and the fourfold sites represent the tetrahedral holes. The fourfold sites are split, for convenience, into two twofold sets. The axial ratio, c_0/a_0 , of the hexagonal unit cell may be varied as desired.

The program has been debugged, and its application to sputtering in copper and germanium is in progress. Extension to zinc will be made soon.

2.2 EXAMINATION OF A THEORETICAL MODEL OF ANISOTROPIC SPUTTERING AT HIGH ENERGIES BASED ON FOCUSING CHAINS

C. Lehmann

Bombardment of single crystal surfaces with ions results in ejection of atoms along low-indexed crystallographic directions. Until recently, available theories⁷ of sputtering employed fitting parameters in order to explain experimental data and

⁷See literature citations in ref 8.

did not account for the anisotropy of this effect. Thompson⁸ was the first to consider crystallographic dependence without the use of fitting parameters. This investigation is based on Silsbee-type⁹ focusing collisions as the elementary event responsible for directional ejection of atoms from the solid.

A more refined and general theory which also is hoped finally to explain the emission ratio (yield) of atoms from single crystals as a function of surface orientation is being developed.

The events leading to ejections of atoms can be separated into two steps: (1) the history of the incoming ion, initiating focusing collision sequences at certain distances away from the surface, and (2) the history of an initiated focusing collision sequence toward the surface, eventually resulting in the ejection of an atom from the surface. The first step involves the question of the mean free path of a primary being shot onto an arbitrary crystal surface. This question is closely related to the problem of channeling.¹⁰ At this time, neither reliable experimental data nor sufficient theoretical calculations are available about anisotropic penetration of an energetic primary into a solid. Therefore, the lattice is considered as random for the history of the incoming ion. This is assumed to be a good approximation since the anisotropy of the second step presumably is considerably higher.

It is assumed that the primary, of kinetic energy E , having normal incidence (all calculations can easily be extended to oblique incidence), collides with a lattice atom at a distance x from the surface and transfers the energy T to this stationary atom. By this energy release, a small zone (with negligible dimensions in comparison to the range of the focuson) of interstitials and vacancies is created, forming the origin of focusing collisions which start from this zone isotropically in all possible close-packed directions. The probability distribution of focuson ranges along close-packed directions has been discussed recently.¹¹ In the

following, this formalism is used and applied, for reasons of simplicity, to copper ions onto copper.

The number of focusons starting in one of the 12 equivalent close-packed $\langle 110 \rangle$ directions from a radiation-damage zone (in which the energy T has been distributed) and having ranges $\geq nD$ (D is the nearest neighbor distance) is

$$\nu = \frac{1}{12} 1.5 \frac{T}{2E_d} \left[1 - \frac{n}{n_0} \left(1 - \ln \frac{n}{n_0} \right) \right], \quad \begin{aligned} n/n_0 &\leq 1, \\ &= 0, \\ n/n_0 &\geq 1, \end{aligned} \quad (1)$$

where $E_d (\approx 25 \text{ ev})$ is the threshold energy for atomic displacement in the bulk, and $n_0 D$ is the maximum range of a focuson along a close-packed direction. In copper, n_0 is ≈ 100 . Let us suppose that all the focusons start at a distance x away from the surface (Fig. 2.1). The geometrical length of a close-packed chain intersecting the surface in a special direction, which includes the angle ϵ with the direction of incidence, is then αx , where $\alpha^{-1} = \cos \epsilon$. To account for the fact that the last atom in the chain has to have a certain minimum energy E_b ("binding energy" or displacement energy for atoms in the surface) in order to be able to leave the surface, we formally increase the geometrical length αx by a distance βD . This distance is chosen in such a way that the energy loss of the focuson after β collisions equals E_b . Roughly, we set $\beta = E_b / \Delta E$, where ΔE is the energy loss per unit length D along the chain. Thus the number of focusons in a special direction " α " reaching the surface with an energy $\geq E_b$ and therefore being able to leave the crystal is, according to Eq. (1),

$$\nu(\alpha, T) = \frac{1}{12} 1.5 \frac{T}{2E_d} [1 - z(1 - \ln z)], \quad \begin{aligned} z &\leq 1, \\ &= 0, \\ z &\geq 1, \end{aligned} \quad (2)$$

where $z = (\alpha x + \beta D) / 100D$.

Now we have to take into account the distribution $W(x; L)$ of collision lengths x (which depends on the mean free path L) and also the distribution $g(T; E)$ of the energy transfer T . From Eq. (2), we have the number of focusons in a direction α

⁸M. W. Thompson, p 85 in *Proceedings of the Fifth Conference on Ionization Phenomena in Gases*, Munich, 1961, vol I, North Holland Publishing Company, Amsterdam.

⁹R. H. Silsbee, *J. Appl. Phys.* **28**, 1246 (1957).

¹⁰C. Lehmann and G. Leibfried, "Long Range Channeling Effects in Irradiated Crystals?" (this report, Part I).

¹¹G. Leibfried, *J. Appl. Phys.* **30**, 1388 (1959).

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Fig. 2.1. Geometric Relation Between Angle and Length of a Close-Packed Atomic Row from a Cascade at Distance x to the Surface.

which start from a radiation-damage zone of energy T at a distance x from the surface and result in an ejection of a surface atom.

Averaging over the distributions W and g gives (for equal masses)

$$\begin{aligned} \bar{\nu}(E; \alpha, \beta) &= \int_0^E dT \int_0^\infty dx W(x; L) g(E; T) \nu(T, x; E; \alpha, \beta) \\ &= \frac{1}{8} \frac{T}{2E_d} \int_0^\infty \frac{e^{-x/L}}{L} [1 - x(1 - \ln x)] dx, \quad (4) \end{aligned}$$

where the average energy transfer

$$\bar{T} = \int_0^E T g(E; T) dT.$$

These integrals can be evaluated easily for hard-sphere interaction. Here we have

$$W(x; L) = \frac{e^{-x/L}}{L}, \quad (5)$$

where the mean free path L is

$$L(E) = 1/n_0 \pi [R(E)]^2, \quad (5a)$$

in which n_0 is the atomic space density of the lattice and R is the hard-sphere radius. For the calculation of L , the mean free path in a randomized hard-core model is used, where the hard-core radius is the distance of closest approach. The employed interatomic potential is $V(r) = Ae^{-r/a}$, with $A = 22.5$ kev and $a = D/13$. The spectrum of energy transfer for hard spheres is

$$\begin{aligned} g(E; T) &= \frac{1}{E}, \quad 0 \leq T \leq E, \\ &= 0, \quad \text{otherwise.} \end{aligned} \quad (6)$$

With Eqs. (5) and (6), $\bar{\nu}$ in Eq. (4) becomes

$$\begin{aligned} \bar{\nu}(E; \alpha, \beta) &= \frac{1}{16} \frac{E}{2E_d} \left\{ (1 + \mu) + (1 + \rho) \left(\ln \frac{\rho}{\mu} - 1 \right) \right. \\ &\quad \left. - e^\rho [Ei(-\rho) - Ei(-\mu)] \right\}, \quad (7) \end{aligned}$$

where

$$\mu = \frac{100D}{\alpha L},$$

$$\rho = \frac{\beta D}{\alpha L},$$

$$-Ei(-x) = \int_x^\infty \frac{e^{-t}}{t} dt, \quad \infty > x > 0; \text{ see refs 12 and 13.}$$

To calculate finally the yield S (number of sputtered atoms per incident primary), we have to sum Eq. (7) over all close-packed $\langle 110 \rangle$ directions within an fcc lattice which intersect the surface (bkl):

$$S_{(bkl)}(E) = \sum_i \bar{\nu}(\alpha_i; \beta; E). \quad (8)$$

This has been done for a (110), a (100), and a (111) plane, considering: for the (110) surface - four directions with chains of length $2x$ and one direction with a chain of length x ; for the (100)

¹²Eugene Jahnke, Fritz Emde, and Friedrich Lösch, *Tables of Higher Functions*, McGraw-Hill, New York, 1960.

¹³*Tables of Sine, Cosine and Exponential Integrals*, Federal Works Agency, Work Projects Administration, New York, 1940.

surface - four directions with chains of length $\sqrt{2} \cdot x$; and for the (111) surface - three directions with chains of length $\sqrt{3/2} \cdot x$. The yield calculated after Eq. (8) is plotted in Fig. 2.2. For the binding energy E_b of a surface atom, 5 ev (corresponding to $\beta = 1$) has been chosen.

At the present stage of the theory, we have agreement with experimental data to within an order of magnitude, which supports the basic idea that focusing collisions are responsible for aniso-

tropic sputtering. We do not yet get the proper sequence $S^{111} > S^{100} > S^{110}$ indicated by experiments¹⁴ for energies above 1 kev. This is the goal of a more detailed investigation. Also, sputtering events will be included in which primaries differ from target materials.

¹⁴A. L. Southern, W. R. Willis, and M. T. Robinson, "Sputtering Experiments with 1- to 5-kev Ar⁺ Ions," see abstract in Part III of this report.

Fig. 2.2. Yield-Energy Relation for Sputtering from (110), (100), and (111) Surfaces for Cu⁺ onto Cu.

3. Semiconductor Theory

3.1 STATISTICAL MECHANICS OF SEMICONDUCTORS

J. W. Miller¹

H. C. Schweinler

The research reported in a preliminary way² in this Report last year has been published as ORGDP Report K-1526, "An IBM 7090 Program for Computing the Fermi-Level in Non-Metals," by

J. W. Miller. Figures 4 and 5 from that report are reproduced here as Figs. 3.1 and 3.2. In them the position of the Fermi level as a function of temperature is given for silicon and germanium for a

¹Central Data Processing Facility, Oak Ridge Gaseous Diffusion Plant.

²J. W. Miller and H. C. Schweinler, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961, ORNL-3213, p 11.*

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Fig. 3.1. Fermi Level in Germanium with Gallium and Antimony Doping as a Function of Majority Carrier Atoms per Cubic Meter.

Fig. 3.2. Fermi Level in Silicon with Gallium and Antimony Doping as a Function of Majority Carrier Atoms per Cubic Meter.

wide range of donor and acceptor concentrations. A very useful summary rule which results from these detailed calculations is that the position of the Fermi level depends primarily on the temperature and the net number of electrons in the crystal and only secondarily on the combinations of numbers of donors and acceptors from which this net number of electrons is obtained. For further details the report referred to above should be consulted. Copies are available on request addressed to the Office of Technical Services, Department of Commerce, Washington 25, D.C.

3.2 DENSITY FUNCTION MODIFYING CARRIER CAPTURE PROBABILITY IN NONMETALLIC CRYSTALS

H. C. Schweinler

J. W. Miller¹

In the theoretical expressions for the minority carrier lifetime in semiconductors, and in the theory of luminescence of insulating crystals, there enters an expression for the capture probability of a charged particle (electron or hole) at a defect. When the defect itself is charged, the

capture probability may be augmented or reduced by a factor which depends on the increase or decrease of the charge density of the incident particle at the capturing center (as a result of Coulomb attraction or repulsion). The modified incident-particle density at the capturing center is assumed to be proportional to the capture probability. The particle density has been averaged with respect to a Maxwell velocity distribution of the bombarding particles, which is valid in the nondegenerate case.

The function so derived is given as

$$D(\zeta) = \frac{2}{\sqrt{\pi}} \zeta \int_0^\infty \frac{e^{-w}}{e^{\zeta/\sqrt{w}} - 1} dw,$$

and is presented in Table 3.1. It depends on the single variable

$$\zeta = \frac{2\pi z}{K} \sqrt{\frac{m^{**}}{m}} \frac{13.6 \text{ eV}}{kT},$$

involving the relative charge z of the two charged points, the effective mass m^{**} of the moving carrier in the crystal, the dielectric constant K of the crystal, and the absolute temperature T . The experimentally determined cross section may be expressed as this factor, $D(\zeta)$, multiplied by an atomic cross section. This determines (approximately) the variable ζ , from which the relative charge of the capturing center can often be inferred.

Table 3.1. Ratio of Particle Density at Capturing Center to Particle Density at Infinity

ζ	$D(+\zeta)$	$D(-\zeta)$	ζ	$D(+\zeta)$	$D(-\zeta)$
0	1.000000	1.000000	22	0.4176077(-4)	24.824384
1	0.5730042	1.7013834	24	0.1930218(-4)	27.081120
2	0.3329456	2.5897040	26	0.9058655(-5)	29.337868
3	0.1959816	3.5811192	28	0.4311674(-5)	31.594622
4	0.1167588	4.6302756	30	0.2079304(-5)	33.851378
5	0.07034540	5.7122414	32	0.1015067(-5)	36.108135
6	0.04282779	6.8131030	34	0.5012264(-6)	38.364894
7	0.02633057	7.9249850	36	0.2501667(-6)	40.621651
8	0.01633680	9.0433704	38	0.1261265(-6)	42.878410
9	0.01022344	10.1656362	40	0.6419729(-7)	45.135168
10	0.6449463(-2) ^a	11.290241	46	0.8918711(-8)	51.905443
11	0.4099585(-2)	12.416271	50	0.2488133(-8)	56.418960
12	0.2624561(-2)	13.543175	56	0.3861689(-9)	63.189235
13	0.1691612(-2)	14.670622	60	0.1150708(-9)	67.702752
14	0.1097269(-2)	15.798406	65	0.2615286(-10)	73.344648
15	0.7160575(-3)	16.926404	70	0.6139716(-11)	78.986544
16	0.4699712(-3)	18.054537	75	0.1484817(-11)	84.628440
17	0.3101412(-3)	19.182756	80	0.3690431(-12)	90.270336
18	0.2057304(-3)	20.311032	85	0.9407581(-13)	95.912232
19	0.1371460(-3)	21.439342	90	0.2455284(-13)	101.55413
20	0.9185772(-4)	22.567676	95	0.6550372(-14)	107.19602
			100	0.1783875(-14)	112.83792

^aThe notation $x(-y)$ means $x \cdot 10^{-y}$.

4. Critical Simplices in Brillouin Zones

H. C. Schweinler

The behavior of the energy as a function of propagation vector in the vicinity of the Brillouin zone surface is well known from the work of Wigner and von Neumann.¹ It leads to energy gaps and a vanishing group velocity at the Brillouin zone surface. The origin of these phenomena is the degeneracy of two states differing in propagation vector by 2π multiplied by a reciprocal lattice vector.

¹E. P. Wigner and J. von Neumann, *Physik. Z.* **30**, 467 (1929).

It is believed to be an essentially new discovery that there are other points and lines in the Brillouin zone for which (in the free-electron approximation) states of the same energy have propagation vectors differing by 2π multiplied by a reciprocal lattice vector. The reduced propagation vectors to which these latter are congruent are called "critical simplices"; a critical 0-simplex is a point in the reduced Brillouin zone; a critical 1-simplex is a line in the reduced Brillouin zone; and a critical 2-simplex is a plane in (possibly entirely within) the reduced Brillouin zone.

Table 4.1. Critical 0-Simplices for a Square Lattice of Points

Critical Simplex	Multiplicity	Congruent Points in Extended Zone	Squared Length	Symmetry ^a
(1/6, 1/6)	3	(7/6, 1/6), (1/6, 7/6), (-5/6, -5/6)	50/36	σ_d
(0, 1/4)	3	(-1, -3/4), (1, -3/4), (0, 5/4)	25/16	σ_v
(0, 1/3)	3	(0, -5/3), (-1, 4/3), (1, 4/3)	25/9	σ_v
(1/4, 1/4)	3	(1/4, -7/4), (5/4, 5/4), (-7/4, 1/4)	50/16	σ_d
(3/14, 5/14)	3	(-11/14, -23/14), (-25/14, 5/14), (17/14, 19/14)	650/196	ϵ
(3/10, 3/10)	3	(-17/10, -7/10), (-7/10, -17/10), (13/10, 13/10)	338/100	σ_d
(1/6, 3/6)	4	(7/6, 9/6), (-11/6, 3/6), (7/6, -9/6), (-11/6, -3/6)	130/36	σ_h
(0, 1/4)	4	(1, -7/4), (2, 1/4), (-1, -7/4), (-2, 1/4)	65/16	σ_v
(1/14, 3/14)	3	(-27/14, -11/14), (15/14, -25/14), (29/14, 3/14)	850/196	ϵ
(1/10, 1/10)	4	(-19/10, -9/10), (-9/10, -19/10), (1/10, 21/10), (21/10, 1/10)	442/100	σ_d
(0, 1/8)	3	(0, 17/8), (-1, -15/8), (1, -15/8)	289/64	σ_v

^aSchoenflies notation.

In some cases, three and four states of the same energy in the free-wave approximation are congruent to the same critical 0-simplex. This leads to a different form of secular equation and a different analytical form of the energy surface from that studied by Wigner and von Neumann. From the 0-simplices as base, 1-simplices can be erected which have these same properties, now as a continuous function of the free coordinate of the propagation vector.

In the case of the square lattice of points in real space, leading also to a square lattice of points in reciprocal space, the critical simplex closest to the origin, other than those of Wigner and von Neumann, is $(\frac{1}{6}, \frac{1}{6})$ and its symmetric equivalents. This simplex is congruent to the three wave vectors $(\frac{1}{6}, \frac{1}{6})$, $(\frac{1}{6}, \frac{7}{6})$, and $(-\frac{5}{6}, -\frac{5}{6})$; each of these is at squared distance $\frac{50}{36}$ from the origin in the extended Brillouin zone. The secular equation factors into a quadratic and

a linear term when the wave vector (assumed to be close to the above critical 0-simplex) lies along the line $[1, 1]$, but does not factor otherwise. It is evident from this brief discussion that a new algebraic structure of the energy surface in the vicinity of this point has been found.

The first 11 critical 0-simplices, some of multiplicity 3, as is that discussed above, and others of multiplicity 4 are given in Table 4.1. Hundreds of critical 0-simplices have been located with the help of J. W. Miller of the ORGDP Central Data Processing Facility.

Each of these 0-simplices in the plane gives rise to a 1-simplex in space, and the intersection of 1-simplices gives certain 0-simplices in space. The space problem for simple cubic, body-centered cubic, and face-centered cubic lattices of points is under active investigation and numerical computation, but no results are available for publication.

5. A Possible Experiment to Study Radiation Damage by the Mössbauer Effect

J. H. Barrett

Although the Mössbauer effect has recently found widespread use as a research tool in physics,¹ this tool has had little application to the studies of defects in solids. Most methods of studying defects in solids, such as electrical resistivity measurements, are very insensitive to the detailed nature of the defects. A few methods of study, such as spin resonance, are sensitive to the details of the structure. In the hope of adding another method of making detailed studies, the following experiment is suggested.

The general requirement for the existence of the Mössbauer effect for a given element is the existence of a stable isotope which has a low-lying nuclear level (less than 100 keV above the ground state) that can be suitably excited. For the present experiment, one other requirement must be added; namely, that the element must also have a stable isotope with a mass number one unit lower. Among the elements that satisfy these two criteria are Fe, Ni, Zn, Ge, Sn, and several rare earths. This group of elements includes one or more examples of all the simpler crystal structures: face-centered cubic, body-centered cubic, diamond, and hexagonal. Because the Mössbauer effect has been studied so extensively in Fe⁵⁷, this isotope will be used as an example in the discussion below.

The proposed experiment would generate the Mössbauer effect by thermal-neutron capture:

The 14.4-keV gamma ray is the one for which the Mössbauer effect is observed. As the higher-energy gamma rays are emitted, they will impart a recoil energy to the neutron-absorbing nucleus, causing it to be displaced from its original lattice position.² The detailed nature of the defect structure produced may affect the energy of the 14.4-keV gamma ray, which is emitted much later than the high-energy gamma rays. The following is an item-by-item analysis of the feasibility of observing the influence of the defect structure on the Mössbauer radiation.

1. A 14.4-keV gamma ray will have a very short mean free path in iron. Within a depth below the surface equal to this mean free path, a 10-cm² iron foil in a flux of 10⁷ thermal neutrons/cm² sec⁻¹ will make about 4 × 10⁴ captures/sec.

2. Recent work³ on the gamma-ray cascades following thermal-neutron capture by Fe⁵⁶ indicates that 50% or more of these cascades will contain the 14.4-keV gamma ray. Items 1 and 2 will yield an acceptable counting rate.

3. In obtaining a thermal-neutron source near a nuclear reactor, there will be a very high gamma-ray background. The effects of the background can be minimized by using a very thin scintillation crystal, a krypton proportional counter, or possibly a semiconductor counter. The background problem can likely be overcome.

4. The high-energy gamma rays will be emitted in a time $\approx 10^{-13}$ sec. Following these high-energy gamma-ray emissions the average recoiling

¹A recent review may be found in: Hans Frauenfelder, *The Mössbauer Effect*, Benjamin, New York, 1962.

²H. C. Schweinler, p 63 in *Chemical Effects of Nuclear Transformations*, vol I, IAEA, Vienna, 1961.

³N. F. Fiebiger, W. R. Kane, and R. E. Segel, *Phys. Rev.* **125**, 2031 (1962).

Fe^{57m} nucleus will have a velocity $\approx 3.5 \times 10^6$ cm/sec. If its range is taken to be ≈ 50 Å, its flight time $\approx 3 \times 10^{-13}$ sec.

5. After the recoiling Fe^{57m} nucleus has come to the end of its flight path, it will still have a few electron volts of energy. Treating this energy as a very localized high temperature and assuming a lattice thermal conductivity of 5×10^{-3} w $\text{cm}^{-1} (\text{°C})^{-1}$, one finds the local temperature will decay to within 10°C of the surroundings in a time $\approx 2 \times 10^{-10}$ sec. Also the occurrence of the Mössbauer effect in the usual way by electron capture of Co^{57} indicates that the thermalization time is less than the half-life of Fe^{57m} , 10^{-7} sec. Items 4 and 5 indicate that the Fe^{57m} nucleus will be stopped and thermalized before emitting the 14.4-keV gamma ray. Therefore, the Mössbauer effect should occur.

6. Since the recoiling Fe^{57m} nucleus will have a few hundred electron volts of energy,² it will probably create several interstitial-vacancy pairs. The recoiling nucleus itself may wind up in an interstitial position, or it may undergo a replacement collision, leaving it at a regular lattice site. If the Fe^{57m} nucleus comes to rest at a normal lattice site, the experiment will not yield any information about defects, although the Mössbauer effect should be observable. If it comes to rest at an interstitial position, the experiment should yield information about the nature of this defect. It is impossible to make a reliable estimate of the probability that the recoiling Fe^{57m} nucleus will end up in an interstitial position. An unfavorably low value of this probability seems to be the only factor that is likely to prevent this

type of experiment from yielding useful information about the nature of an interstitial. In order to have the Fe^{57m} nucleus remain in the interstitial position until it emits the 14.4-keV gamma ray, it may be necessary to have the neutron-absorbing foil at a low temperature.

7. This final item concerns the way in which the interstitial position of the Fe^{57m} might alter the Mössbauer effect. Ludwig and Woodbury⁴ suggest on the basis of spin resonance studies that transition metals as interstitials in silicon transfer their 4s electrons to the 3d shell. When a transition element atom is squeezed, this seems likely to happen; it will be assumed to happen for an iron atom as an interstitial in iron. Detailed studies^{5,6} of the Mössbauer radiation from Fe^{57} indicate that there is a chemical shift and a change in the hyperfine splitting if there is a $3d^8$ electronic configuration around an Fe^{57m} nucleus rather than the regular $3d^7 4s$ configuration of metallic iron. Also, the interstitial arrangement in iron might be a split interstitial similar to that of the face-centered cubic metals. If this is the arrangement, there would be a quadrupole interaction, which would be a further observable effect.

⁴G. W. Ludwig and H. H. Woodbury, *Phys. Rev.* **117**, 1286 (1960).

⁵L. R. Walker, G. K. Wertheim, and V. Jaccarino, *Phys. Rev. Letters* **6**, 98 (1961). This is reprinted on p 236 of ref 1.

⁶G. K. Wertheim and D. N. E. Buchanan, p 130 in *Proceedings of the 2nd International Conference on the Mössbauer Effect* (ed. by D. M. J. Compton and Alan H. Schoen), Wiley, New York, 1962.

6. Electron Cross Sections for Atomic Displacements

O. S. Oen.

In a previous report¹ a calculational program was outlined for computing the number of atomic displacements produced by elastic collisions between fast electrons and atomic nuclei. At that time a FORTRAN program had been written for an IBM 7090 computer which allowed one to compute the primary displacement cross section using a sharp displacement threshold, for arbitrary electron energy, but limited to elements of low atomic number ($Z < 27$). This latter restriction resulted from using the McKinley-Feshbach version of the Mott series to describe the electron-nucleus scattering. A new FORTRAN program has been written which evaluates numerically the Mott series while computing the primary displacement cross section. The method used to calculate the Mott series is that of Doggett and Spencer.² With the use of this new program, which is good for arbitrary Z , the primary displacement cross sections have been calculated for $Z = 29, 47, 50, 79, 82$, and 92 , using several sharp displacement threshold energies. For uranium, Fig. 6.1 shows that an error of a factor of 3 may arise from using the McKinley-Feshbach formula. For an element of low Z , such as copper, the two formulations differ by less than 10%. An unexpected, interesting feature of the Mott formulation is the pronounced maximum found in the primary displacement cross section. This maximum occurs in a highly relativistic region and, therefore, cannot be explained within the framework of nonrelativistic mechanics,

as were the cases found by Compton and Arnold.³ In general, this relativistic-type maximum becomes less pronounced for elements of lower Z . It is planned to extend these calculations to include the cascade of displacements initiated by the primary knock-on.

Fig. 6.1. Primary-Displacement Cross Section in Uranium as a Function of Energy, by using a Sharp Displacement Threshold of 25 ev.

¹O. S. Oen, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, p 9.

²J. A. Doggett and L. V. Spencer, *Phys. Rev.* **103**, 1597 (1956).

³W. D. Compton and G. W. Arnold, *J. Appl. Phys.* **31**, 621 (1960).

Part II. Crystal Physics

M. K. Wilkinson

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7. X-Ray Diffraction

7.1 SINGLE CRYSTALS GROWN FROM SMALL DROPLETS¹

M. C. Wittels F. A. Sherrill
J. O. Stiegler²

Single-crystal droplets of Au, Cu, Ag, Pb, In, Cu₃Au, and Ge have been grown in vacuum by two simple methods. In each case graphite crucibles were employed and served as a point contact so that the droplets could solidify in the absence of a containment shell, free of mechanical and thermal stresses. The crystals approximate spheres 50 to 600 μ in diameter and are thus ideally suited for x-ray diffraction studies in transmission. Although the surface-tension properties of the materials were important to the successful growth characteristics, it was also observed that exclusion of foreign nucleation catalysts and elimination of mechanical and thermal stresses were equally as significant.

7.2 RADIATION EFFECTS IN URANIUM-DOPED ZIRCONIA³

M. C. Wittels J. O. Stiegler²
F. A. Sherrill

A new study concerning the mechanism of the radiation-induced phase transformation, monoclinic to fcc, in ZrO₂ is presented. The most important aspect of these experiments was the effort to separate the effects of fast neutrons, fission fragments, and uranium chemical impurities. This was accomplished by (1) carefully

doping chemically pure ZrO₂ samples with selected concentrations of enriched and depleted uranium, (2) irradiating these samples in closely monitored thermal-neutron and fast-neutron environments and correlating the phase changes observed with the specific particle events, (3) irradiating ZrO₂ that was obtained by oxidizing Zircaloy-2 which was free of fissionable impurities with a fast-neutron dose greater than 6×10^{20} neutrons/cm², and (4) subjecting a large group of reactor-grade zirconium and its alloys, selected at random, to neutron activation analysis for U and Th impurities.

The results of the investigation indicate that (1) fission fragment fluxes associated with 10^{15} – 10^{16} fissions/cm³ produce a monoclinic to fcc phase change in ZrO₂, and the presence of U in ZrO₂ in concentrations less than 0.38 wt % does not act as a nucleation or stabilizing atom in the process, (2) the stability of the phases of ZrO₂ under reactor irradiation depends entirely upon the action of a fission fragment flux of sufficient magnitude, and where conflicting data have been reported it is believed that an unknown presence of fissionable impurities would resolve these differences, (3) fast neutrons, solely, cannot induce the phase transition at doses up to 6.3×10^{20} neutrons/cm², and (4) in 21 analyses for U and Th in randomly selected reactor-grade zirconium and its alloys, fissionable impurities were found in 20 cases. Natural-uranium concentrations varied between 1.8 and 6.3 ppm, and thorium concentrations varied between 0.29 and 50.0 ppm.

Two different models are given to explain the mechanism of fission fragments in producing the phase transition. Both models require the collapse of small regions due to the pressures exerted by interstitials. In one case the action takes place at the beginning of the range of the fission fragment through an ionization displacement process, and in the other instance the process occurs near the end of the fission fragment where

¹Abstract of article to be submitted for publication.

²Metals and Ceramics Division.

³Abstract of paper to be published in the *Journal of Nuclear Energy*.

elastic collisions predominate. The effects of temperature are not clear and are not of prime importance, but the radiation stability of the fluorite structure of ZrO_2 , as in the case of UO_2 , appears to be significant.

7.3 A VERSATILE LANG CAMERA

M. C. Wittels F. A. Sherill
G. F. Fielder

Development work is being furthered in an effort to acquire a versatile Lang⁴ camera in which imperfections arising from several sources may be observed in nearly perfect crystals. It is believed that the instrument finally developed will permit one to observe as-grown dislocations, mechanically and thermally induced dislocations, foreign inclusions, small-angle tilts, and structural defects produced by various sources of radiation. The instrument being developed utilizes some of the aspects put forward by Webb⁵ and Schwutke⁶ in their studies of lattice imperfections, but we have modified the instrumentation to include a portable crystal rocker for measuring rocking-curve line-widths of nearly perfect crystals.

Photographs of the apparatus are shown in Figs. 7.1–7.3. A conventional x-ray source (Ge-XRD-5) with a silver anode for thin-crystal transmission studies is utilized together with a diffractometer and scintillation counter for crystal alignment purposes. As shown, the anode-to-crystal distance has been increased to 92 cm, and a slit $2.5 \text{ cm} \times 2.5 \text{ cm} \times 0.038 \text{ mm}$ is employed to further collimate the x-ray beam. Ilford x-ray film or G-5-type nuclear plates are contained in the light-tight easily removable film cassette that can be translated on a track along the line of the diffracted x-ray beam. Similarly, the direct-beam screen and scattering catcher has translatable movements with three degrees of freedom.

Conventional small-crystal goniometers are mounted on the center of the camera table and have a screw adjustment for vertical motion. For turning the crystal slowly about a vertical axis into the diffracting position, a gear reduction of

⁴A. R. Lang, *Acta Met.* **5**, 358 (1957).

⁵W. W. Webb, *Direct Observations of Imperfections in Crystals*, pp 29–76, Interscience, New York, 1962.

⁶G. H. Schwutke, *J. Electrochem. Soc.* **109**, 27 (1962).

Fig. 7.1. Lang Camera with Crystal Rocker.

Fig. 7.2. Lang Camera Closeup Showing Screen and Film Holder.

1800:1 is utilized, and this same gear system, when coupled with the synchronous 1-rph motor, permits an angular rock of 12 sec/min. In nearly perfect crystals, where the half-width at maximum intensity at low index reflections is often less than 30 sec, it is desirable, if not necessary, to possess these slow motions. The rocking motor is portable and is decoupled from the table by

means of a magnetic base mount when a translation motion is used.

The translating table is driven by a center-mounted screw over a span of 2 in., and the table motion is reversed through microswitches attached to adjustable stops so that the translation span can be limited to the crystal width.

Fig. 7.3. Portable Crystal Rocker for Lang Camera.

The translation of the crystal is vital to the Lang technique in two respects: (1) the motion should be synchronous, and (2) the motion should be linear to within at least a few seconds of arc. Failure to obtain either or both of these features greatly hampers the effort to obtain clear diffraction topographs without obscurities in reasonable times. Present work with the development of the instrument is aimed at perfecting these features.

7.4 THE 1961 POSTANNEALING OF GRAPHITE-STORED ENERGY IN THE ORNL GRAPHITE REACTOR

M. C. Wittels

Following the September 1961 annealing of the ORNL Graphite Reactor, graphite cores were taken from the three main areas of interest in the moderator stack, and stored energies were measured

by the radiation-calorimetric technique. The selection of these areas was predetermined by the 1960 discoveries of stored energy in the extreme corners of the fuel zone and in the first reflector rows, as well as by the peak temperatures reached during the September 1961 anneal.

The measured graphite stored energies are given in Table 7.1 and are largely self-explanatory. These data are also plotted in Fig. 7.4 as stored energy distributions along the channels; however, it should be noted that the energies released as graphically shown were measured to somewhat different peak temperatures (Table 7.1) and are therefore not strictly comparable; nevertheless, they are meaningful. Figure 7.5 shows (a) the 140°C isotherm reached during the 1961 annealing effort in a plane 7 ft 6 in. west of the east graphite face, (b) the inner periphery in this plane of the preannealing stored energy region, and (c) the channels selected for stored energy determinations. The shaded area (Fig. 7.5) indicates the approximate area of successful annealing in this same plane achieved as a result of the 1961 annealing operation. Since the temperatures further toward the center of the reactor core were appreciably higher during the annealing operation (>200°C), it is apparent that in the central region the integrated stored energy up to 250°C was reduced to near zero as a result of the 1961 operation.

Fig. 7.4. Stored-Energy Distribution Along Fuel Channels.

Table 7.1. Stored-Energy Data – October 1961

Channel	Distance from East Edge (ft)	Energy (cal/g)	Maximum Temperature (°C)
Corner Fuel Channels			
2980	3	17.2	242
2980	6	40.7	290
2980	10	45.4	296
2980	14	33.9	268
2957	5	43.7	293
2957	9	58.6	315
2957	11½	54.7	309
0757	4	39.5	278
0757	7	56.9	316
0757	11½	50.4	301
0780	3	16.8	244
0780	6	59.8	296
0780	9	41.4	287
Lateral Fuel Channels			
3167	4	27.4	256
3167	9	42.4	262
3167	11½	35.6	256
2055	4	31.3	279
2055	7	34.7	273
2055	11	36.3	273
0569	4	30.4	266
0569	7	31.4	267
0569	10	32.7	269
1982	4	16.9	249
1982	7	14.9	247
1982	10	18.9	250
Reflector Channels			
3467	5	2	237
3467	8	7.0	239
3467	11½	2	237
3567	5	0	237
3567	8	0	237
3567	11½	0	237
3055	5	7.9	237
3055	8	13.0	238
3055	11½	14.4	238
3255	5	0	237
3255	8	0	237
3255	11½	0	237
3453	5	0	237
3453	8	0	237
3453	11½	0	237

Fig. 7.5. Front-Face View of Stored-Energy Status 7 ft 6 in. from East Graphite Face.

It is clear from the stored-energy measurements in the *lateral fuel channels* close to the 140°C isotherm (Fig. 7.5) that the spontaneously releasable stored energy in the *lateral* areas has been largely reduced, so that the thickness of the areas is probably no greater than 12 in. and may possibly approach zero on the south and bottom sides of the fuel zone.

The accumulation of stored energy in the corner fuel channels continues to grow at about $2\frac{1}{2}$ cal g⁻¹ yr⁻¹ to 250°C, and at present is of a magnitude to cause an adiabatic rise in those regions to 350–375°C, if the energy is released under ideal adiabatic condition. The total volume of graphite in these affected areas is about 7% of the volume of the entire graphite stack, and therefore contains about 4.5 Mwhr of adiabatically releasable stored energy at present.

In assessing the success of the 1960 and 1961 annealing operations the fact should not be overlooked that the predicted 1960 stored-energy region has been successfully annealed to a high degree. In addition, it has once again been shown that it was virtually impossible to heat the corner regions of the fuel zone to annealing temperatures, even under the contrived conditions of reverse airflow and blocked airflow with scrambled conditions from high temperature.

The growth of significant amounts of stored energy in the extreme corners of the fuel zone, even out to the very last slug rows, is still not adequately understood. For many winter months the temperatures in these channels is considerably lower than 30°C, and presumably the damage rate at 10°C might be three times greater than the rate at 30°C, the lowest temperature at which important amounts of data are available. Since the fast neutron flux >1 Mev in these extreme fuel corners is probably below an order of magnitude lower than that required by accepted radiation-damage theory to account for the damage produced, it appears that present radiation theory is inadequate in this regard. As a matter of speculation it is felt that some of the stored-energy problems in other air-cooled graphite reactors (e.g., Windscale, BEPO, and BR-1) might have been due to stored-energy accumulations at the extreme edges of the fuel zones in these reactors, where it previously could have been easily overlooked.

Since the airflow in the reflector channels of the ORNL Graphite Reactor has been blocked, more coolant gas now passes through the entire fuel

zone, and it therefore operates at a somewhat lower temperature during regular operation. Under these conditions stored energy would accumulate at a somewhat higher rate in the reactor during future years. Development work to provide a means of annealing the peripheral stored-energy regions of the reactor is proceeding satisfactorily, and it is hoped that the end result will furnish a flatter temperature distribution in the reactor core for normal operation as well as provide a sufficient thermal configuration for successful annealing.

7.5 X-RAY OBSERVATIONS OF NEARLY PERFECT COPPER SINGLE CRYSTALS⁷

M. C. Wittels F. A. Sherrill
F. W. Young, Jr.

It is well known that metal crystals are generally imperfect and that their dislocation densities usually exceed $\sim 10^6/\text{cm}^3$, thereby making individual dislocations unresolvable when utilizing the Lang⁸ technique. However, recent etch-pit studies⁹ have indicated that large copper single crystals could be grown with a much higher degree of perfection than had been previously realized for any metal crystals except perhaps whiskers. This paper is a presentation of the initial x-ray diffraction studies of some of these highly perfect⁹ copper crystals.

We have utilized the Lang method for observing dislocation concentrations in bulk samples by employing techniques similar to those described by Webb¹⁰ and Schwuttke.¹¹ As an adjunct to the Lang camera, we have also adapted a portable crystal rocker which permits the measurement of line half-widths in seconds of arc.

The optimum contrast conditions for observing dislocations through the Lang technique require that $\mu t \approx 1$, where μ is the linear absorption

⁷M. C. Wittels, F. A. Sherrill, and F. W. Young, Jr., *Appl. Phys. Letters* **1**, 22 (1962).

⁸A. R. Lang, *Acta Met.* **5**, 358 (1957).

⁹F. W. Young, Jr., *Bull. Am. Phys. Soc.* **7**, 215 (1962).

¹⁰W. W. Webb, *Direct Observations of Imperfections in Crystals*, pp 29–76, Interscience, New York, 1962.

¹¹G. H. Schwuttke, *J. Electrochem. Soc.* **109**, 27 (1962).

coefficient, and t is the crystal slice thickness. Since we used Ag $K\alpha$ radiation, it was necessary to thin crystals to approximately 0.002 in. without introducing imperfections and strains. Needless to say, this is a very difficult task with a soft metal such as copper, and considerable caution must be exercised. This was successfully accomplished by first cutting wafers with an acid saw¹² and then slowly electropolishing to the proper thickness. The orientation selected for the x-ray transmission studies was a (110) face with diffraction from (111) planes which were perpendicular to the crystal surface.

Some of the Lang diffraction topographs obtained from these crystals are shown in Fig. 7.6. It is evident from these topographs that large areas of the crystals are completely free of dislocations. It is also apparent that dislocations which lie in or close to the plane of the wafer are strikingly evident and, moreover, that the strain field surrounding the dislocation core in copper can be as high as $50\ \mu$. Etch-pit counts⁹ of dislocations in random slices from the same single-crystal ingot give a good correlation between the two methods, and confirm the proposal⁹ that crystals

with dislocation concentrations as low as $5 \times 10^2/\text{cm}^3$ are possible in copper.

As a further guide to the study of perfection of these copper crystals, rocking curves were run in reflection from (111) surfaces of large crystal slabs approximately $1 \times \frac{1}{4} \times \frac{1}{4}$ in. The same incident x-ray beam was utilized as for the Lang geometry, namely Ag $K\alpha$ radiation collimated to give a total beam divergence of 15 sec. Under these conditions a clear separation of the $K\alpha$ doublet was obtained, and the measured width of $K\alpha_1$ at half maximum intensity was 34 sec (Fig. 7.7). Although this is not the classical method for rocking-curve measurements, which requires strictly monochromatic radiation in the double-crystal spectrometer arrangement,¹³ it at least gives an upper limit of the absolute half-width. Furthermore, we feel it is probably quite close to the absolute half-width, since we have measured half-widths on germanium (111) as low as 23 sec with the same geometry. The etch-pit determination of dislocations on this same crystal indicated the presence of $\sim 3 \times 10^2$ dislocations/ cm^3 . If this is the case, then probably some type of dislocation or imperfection other than these dis-

¹²F. W. Young, Jr., and T. R. Wilson, *Rev. Sci. Instr.* **32**, 559 (1961).

¹³R. W. James, *The Optical Principles of the Diffraction of X-Rays*, Bell, London, 1950.

Fig. 7.6. Lang Diffraction Topographs of Three Copper Crystals. Orientation as shown. 10X.

locations contributes to the line breadth observed in this crystal. It might possibly be due to small vacancy clusters or loops resulting from the collapse of such clusters.

It is hoped that further refinement in the use of the Lang technique as well as use of the double-crystal spectrometer will aid in the determination of imperfections in copper.

Fig. 7.7. Rocking Curve ($\text{Ag K}\alpha_1$) of Copper (111).
Peak intensity approximately 8700 counts/min.

8. Electron Microscopy

8.1 FISSION-FRAGMENT TRACKS IN METAL FILMS¹

T. S. Noggle

J. O. Stiegler²

Fission-fragment tracks observed in electron microscope studies of platinum and palladium films are interpreted in terms of thermal processes involving melting and vaporization. The results require that the energy lost by the fragment be converted into thermal energy over the entire volume of the track in a time short enough that little of it is conducted away. A model in which free electrons disperse the energy over this volume is used to account for the widths and structures of the tracks.

8.2 FISSION-FRAGMENT TRACKS IN THIN FILMS OF UO_2 ³

T. S. Noggle

J. O. Stiegler²

Surface effects in UO_2 films produced by individual fission events which can be studied with the electron microscope offer promise of giving increased insight into the interaction of fission fragments with solids. Experimental techniques are described in which it is possible to identify the class of fragment giving rise to a particular length of track, the direction of travel of the fragment in producing the track, and the depth of the fragment below the free surface at points along the track. The results obtained with these techniques are correlated with the structures observed.

The proposed mechanisms of energy loss of fission fragments are discussed and related to the studies of track structure. Results suggest that electron excitation and ionization energy loss are dominant in determining track registration. Finally, a new mechanism by which ionization energy may be converted into lattice damage is described.

8.3 NITROGEN ION BOMBARDMENT OF THIN PLATINUM FILMS⁴

J. O. Stiegler²

T. S. Noggle

Previous studies⁵ of thin, polycrystalline films of platinum and palladium irradiated with fission fragments revealed track structures that indicated material had been displaced, melted, or vaporized by the passage of the fragments through the films. The features of the tracks were explained⁵ in terms of a model developed by Ozeroff,⁶ in which free electrons disperse the energy lost by the fission fragment over the entire track volume before it appears as thermal energy.

In this model the fission fragment excites electrons in a cylindrical cloud about 50 to 75 Å in diameter; this transfers an average of ~90 eV to each electron. The excited electrons move into the lattice, collide with conduction electrons, and reach conduction energies during the time they move ~10 Å. Within an additional 100 to 200 Å they reach equilibrium with the lattice. The energy lost by the fragment, therefore, appears as thermal energy over a region extending 100 to 200 Å from the fission fragment's path.

¹Abstract of published paper: *J. Appl. Phys.* **33**, 1726 (1962).

²Metals and Ceramics Division.

³Abstract of paper published in *Am. Soc. Testing Mater. Spec. Tech. Publ.* **306**, 47-63 (1961).

⁴J. O. Stiegler and T. S. Noggle, *J. Appl. Phys.* **33**, 1894 (1962).

⁵T. S. Noggle and J. O. Stiegler, to be published in the *Journal of Applied Physics*.

⁶J. Ozeroff, KAPL-205, pp 22-32 (1949).

The results of observations of a series of palladium films⁵ varying in thickness between 25 and 400 Å were in agreement with this model. In the thinnest films, tracks of light contrast were observed having widths of 200 to 400 Å, indicating that material had been removed from this region, probably due to vaporization. Films of intermediate thickness showed dark tracks, indicating melting followed by rapid cooling of the track region. Thicker films failed to show any evidence of tracks. These results were interpreted in terms of energy density within the track region. Reflection of the low-energy electrons by the potential barrier of the free surface confined them to the films and raised the energy density in the thinnest films high enough to cause complete vaporization. Only melting was possible in films 100 Å thick, and in films greater than 200 Å thick the energy density was not high enough to cause melting.

In order to test these ideas, a series of cyclotron irradiations of platinum films (<25 Å thick) with 24-Mev nitrogen ions was carried out. The energy loss rate of 24-Mev nitrogen ions in platinum is about 500 to 600 ev/Å, compared with 4000 ev/Å for typical fission fragments.⁷ As a consequence, the energy density in the platinum films irradiated with the nitrogen ions should not be high enough to cause vaporization; however, uncertainty as to the exact film thickness leads to uncertainty as to whether melting should occur. On the basis of these considerations along with the observations and interpretation of the fission-fragment tracks in metal films,⁵ one would expect to see broad tracks indicative of melting if tracks can be observed as a result of the ion bombardment.

The platinum films were formed by evaporation onto the surfaces of carbon films (on the order of 200 Å thick) which had previously been evaporated onto fresh cleavage surfaces of NaCl. The irradiations were carried out in the ORNL 63-in. cyclotron. Films were irradiated on NaCl substrates positioned with the plane of the film

parallel to the direction of the ion beam, which was diffused by passing it through two thin nickel foils prior to striking the specimen. The initial energy of the beam was 28 Mev, and it was degraded about 2 Mev in passing through each of the foils. After the irradiation the composite platinum-carbon film was removed from the NaCl and mounted on standard electron microscope grids for examination.

A transmission electron micrograph of a typical region of an irradiated film is shown in Fig. 8.1. For comparison a micrograph of an identical film after fission-fragment irradiation⁵ is shown in Fig. 8.2. The tracks in both cases are light, indicating that material has been removed from the track region. The tracks formed by fission fragments are of considerably higher contrast, suggesting that only partial removal of material occurred with the nitrogen ion irradiation. In addition, the tracks produced by the nitrogen ions are extremely narrow, 25 to 50 Å wide, compared with fission-fragment tracks nearly 400 Å wide. The occasional broad, contrasty tracks observed in the nitrogen-ion-irradiated film result from the superposition of two or more of the narrow, low-contrast tracks.

The tracks produced by the nitrogen ion irradiation are completely different from the type predicted by the Ozeroff treatment in that they are narrow and light rather than broad and dark. Simple vaporization as a result of dispersal of the energy lost by the ion over the track volume by the free electron cloud cannot account for the observations. Since the volume over which electrons are excited by the nitrogen ions is less than that by fission fragments and also since the average energy transfer is less, one might expect a smaller volume to be ultimately affected by the ions. However, the major factor determining the width of the affected zone is the distance required for the slowed-down (conduction energy) electrons to come to equilibrium with the lattice. This distance is on the order of 100 Å and should not vary for the two cases. The track widths, therefore, are too narrow to be explained on the basis of the Ozeroff theory.

These observations suggest that these tracks are not the result of a thermal effect. Quite possibly they occur through charge interaction between the moving ion and the surface layers of the film. If sufficient electrons are accelerated out of the film by the moving ion, a thin layer

⁷Energy loss rates of nitrogen ions have been measured in nickel by H. L. Reynolds, D. W. Scott, and A. Zucker, *Phys. Rev.* **95**, 671 (1954), and energy loss rates of fission fragments in nickel and gold have been measured by C. B. Fulmer, ORNL 2320 (1959). The figures used were determined by assuming that the loss rate in platinum is about equal to that in gold, and that the ratio of loss rates in gold to nickel for fission fragments is approximately equal to that for nitrogen ions.

Fig. 8.1. Tracks Produced in a Platinum Film <25 A Thick by 24-Mev Nitrogen Ions. 50,000X.

Fig. 8.2. Fission-Fragment Tracks in a Platinum Film <25 A Thick. 50,000X.

at the surface will be left with a net positive charge. Free electrons will move into the region to neutralize the charge imbalance, but the separation of charge will probably be of sufficient duration that the electrostatic repulsion of the remaining positive ions will displace material at the surface, thus producing the light tracks. This process may occur to some extent in thicker films, but the relative disturbance would be too small to produce a detectable contrast effect. Only the broad tracks occurring from thermal effects when the ion is well within the interior of the film would be observed.

These results show that ions as light as nitrogen will leave tracks in very thin metal films. However, tracks left by 24-Mev nitrogen ions in platinum films <25 Å thick are not the result of thermal processes responsible for fission-fragment tracks in thicker metal films. Direct charge interaction may displace sufficient material at a free surface to lead to a detectable contrast in very thin films.

The cyclotron irradiations were carried out under the direction of A. Zucker and M. Halbert of the Electronuclear Division.

8.4 TRANSMISSION ELECTRON MICROSCOPE STUDIES ON SINGLE CRYSTALS OF UO_2

T. S. Noggle

Preliminary observations have been made by transmission electron microscopy of fission-fragment tracks in single crystals of UO_2 . These tracks appear to be limited to the surface regions of the crystal and are not observed in specimens thinned subsequent to irradiation. No structural damage has been identified for the low irradiation doses employed to date; however, it has been found that some reaction occurs between UO_2 and its environment when irradiated in air in a polyethylene capsule. Figure 8.3 shows a prethinned UO_2 crystal irradiated in vacuum, and fission-fragment tracks are visible, but there are no other detectable changes from the unirradiated condition. Figure 8.4 shows a prethinned UO_2 crystal irradiated in air in a polyethylene capsule. The marked change in structure reflects in good part the microcracking arising from a volume

Fig. 8.3. Crystal of UO_2 Irradiated in Vacuum to a Dose of 5×10^{14} fissions/cm³. 100,000X.

Fig. 8.4. Crystal of UO_2 Irradiated in Air in a Polyethylene Capsule to a Dose of 5×10^{14} fissions/cm³. 100,000X.

decrease accompanying the reaction. Lower irradiation doses indicate that material from the environment initially reacts with the UO_2 at discrete points on the surface, giving a coherent phase of higher density. Figure 8.5 shows such a specimen in which the small patches, believed to be reaction sites, show contrast effects associated with strain fields. Figure 8.6 shows several patches containing Moiré patterns, indicating that the material in the patch is crystalline and coherent with the UO_2 . These structures are limited to the surface regions of the crystal and are eliminated completely by a short etch in hot nitric acid. The new, thin regions present after etching show no trace of structures such as are present in Figs. 8.4–8.6, and it is estimated that the depth to which the reaction has progressed in Fig. 8.4 is of the order of $0.1\ \mu$ or less.

A tentative explanation of these observations is that volatile organic material released by the polyethylene during irradiation reacts with the UO_2 to form, in part at least, uranium monocarbide (UC). Moiré fringe spacings of $31.2\ \text{\AA}$

Fig. 8.5. Specimen of UO_2 Irradiated to 5×10^{13} fissions/ cm^3 in Polyethylene Capsule. 50,000X.

Fig. 8.6. Moiré Patterns in Reaction Sites on UO_2 Irradiated to 5×10^{13} fissions/ cm^3 in Polyethylene Capsule. 400,000X.

have been observed in several instances and agree quite well with the spacing expected of the (111) reflection from UC in parallel orientation with UO_2 . The volume decrease associated with reduction and carburization would lead to cracking of the reactant layer in the latter stages of the process.

Although many details of this phenomenon need to be studied before the full significance of the observations will be known, these observations graphically illustrate the effect of environment on the surface integrity of UO_2 during irradiation.

8.5 DISLOCATIONS IN UO_2 ⁸

T. S. Noggle

During the development of techniques for preparing single crystals of UO_2 for electron

⁸To be published in the *Proceedings of the 5th International Congress for Electron Microscopy*, Aug. 29–Sept. 5, 1962, Philadelphia, Pennsylvania.

microscope studies of fission-fragment damage, observations have been made on dislocation structures which reflect an appreciable degree of plasticity at elevated temperatures.

Small platelets of UO_2 on the order of $\frac{1}{2}$ mm in diameter and 10 to 100 μ thick were obtained from UO_2 formed by hydrolysis of UF_4 in a molten salt bath. These platelets were thinned by etching in hot fuming nitric acid to give areas suitable for transmission microscopy. During the course of thinning, a number of crystals developed arrays of etch pits delineating slip lines and slip bands as shown in Fig. 8.7. The orientation of all platelets

showing these etch-pit patterns has proved to be (111), and the slip-line structures are in agreement with the slip system (100) [110] reported for fluorite.⁹ These dislocations were apparently introduced at elevated temperatures, probably during solidification of the molten salt in which they were grown. Electron microscope observations of dislocations in the etch pits as shown in Fig. 8.8 establish the relationship between etch pits and

⁹E. Schmidt and W. Boas, *Kristall Plastizitat*, Springer, Berlin, 1935.

Fig. 8.7. Optical Micrograph in Transmitted Light of (111) UO_2 Crystal. $a-a'$, etc., indicate pairing of rows of etch pits developed on the top and bottom surfaces respectively.

Fig. 8.8. An Electron Transmission Micrograph from Row of Etch Pits in a Slip Line in a (111) UO_2 Crystal.

dislocations in this system. In general, very few dislocations are observed in the electron microscope in (111) crystals, since the etch attack is accelerated at the dislocations terminating in the free surface (giving etch pits), and these regions are etched away before larger areas become thin enough for electron transmission. Dislocations are frequently observed in (110) crystals, which do not develop etch pits. Figure 8.9 shows an example of dislocations observed in a (110) crystal. The curvature of some of the dislocations seen here reflects the immobility of the dislocations at room temperature and indicates that the crystal was stressed during cooling from tem-

peratures where the dislocations were mobile. Attempts to determine the Burger's vectors have been limited to diffraction dark-field observations of regions in which multiple reflections occur, and yield the result $b = k, l$ unspecified. This result is compatible with the $[110]$ slip direction noted above.

Movement of the dislocations has not been observed under normal viewing conditions; however, during attempts to make observations in a region of marginal thinness, the beam was temporarily raised to a high value and rapid generation and motion of dislocations ensued. Figure 8.10 is from this area and shows dis-

Fig. 8.9. Dislocations in a (110) UO_2 Crystal Introduced During Growth or by Plastic Deformation During Cooling from Elevated Temperatures.

locations and slip traces generated by their movement. Crystallographic analysis of the structures observed in this region is only in part compatible with a (100) [110] slip system and

suggests that cross slip occurs in this material and that other slip systems may have been active under the ill-defined conditions established in this case.

Fig. 8.10. Dislocations and Slip Traces Induced by Heating of the Electron Beam in a (111) UO_2 Crystal.

9. Spin Resonance

9.1 RELATION BETWEEN MAGNETIC SUSCEPTIBILITY, ELECTRON SPIN RESONANCE, AND OPTICAL ABSORPTION OF THE E'_1 CENTER IN FUSED SILICA¹

R. A. Weeks

E. Sonder

Through a series of irradiations with energetic particles followed by light bleaching and heat treatments of several varieties of fused silica, a one-to-one correspondence is found between an optical band at 2120 Å and an electron spin resonance (ESR) line $g = 2.0006$. A comparison is then made between the concentration of centers in this ESR line determined by an ESR comparison technique, and the total concentration is measured by a magnetic susceptibility method. The results of the comparison show that the primary paramagnetic center and the center which gives the ESR line $g = 2.0006$ are the same. This center has been called the E'_1 center. After appropriate irradiations and subsequent bleaching treatments, the concentration of spins in the E'_1 center was measured by the susceptibility technique. The absorption coefficient and the width at half maximum absorption coefficient were found, and using Smakula's equation appropriate for a Gaussian function, the oscillator strength f was found to be $f = 0.14 \pm 0.04$.

9.2 PARAMAGNETIC SPECTRA OF E'_2 CENTERS IN CRYSTALLINE QUARTZ²

R. A. Weeks

The electron spin resonance spectra of the E'_2 center, a defect of the quartz structure, are in

agreement with the spectra predicted by a spin Hamiltonian, $S = \frac{1}{2}$,

$$\mathcal{H} = \sum_{i=1}^6 (\vec{H} \cdot \vec{g}_{1i} \cdot \vec{S} + \vec{H} \cdot \vec{g}_{2i} \cdot \vec{S}).$$

The i subscript refers to the symmetry operations of the crystal necessary to obtain the six components of each line observed for the magnetic field H parallel to the c axis of the crystal. The eigenvalues of the two g tensors are:

$$\begin{aligned} g_1 (g_{c \text{ axis}} = 2.0008) [g_{11} = 2.0015, \\ g_{\perp} = 2.0006]; \\ g_2 (g_{c \text{ axis}} = 2.0010) [g_1 = 2.0022, \\ g_2 = 2.0005, g_3 = 2.0007]. \end{aligned}$$

Hyperfine interactions are observed with nearby nuclei. On the basis of the intensity ratio of these lines to the unperturbed lines, it is suggested that the interaction is with Si^{29} , $I = \frac{1}{2}$. Interaction of the defect electron with one Si^{29} produces a splitting of $A \approx 412$ gauss (c axis is parallel to the applied field). A comparison of $|\psi(0)|^2$ calculated from this interaction with the value of $|\psi_{3S, \text{Si}} 3+(0)|^2$ indicates that the silicon atom is at the defect site. The remaining interactions are very weak; $A_1 = 1.0$ gauss, $A_2 = 0.8$ gauss, $A_3 \approx 0.5$ gauss (c axis is parallel to the applied field). These lines are interpreted as being due to an Si^{29} in any one of three nearby sites. Assuming a dipole-dipole interaction, the separation of these sites from the defect is ≥ 1.7 Å. A possible interpretation of these results in terms of plausible models is given, and a comparison is made with the magnetic characteristics of another defect of the quartz structure, the E'_1 center.

¹Abstract of paper to be published in the *Proceedings of the 1st International Conference on Paramagnetic Resonance*, July 16–20, 1962, Jerusalem, Israel.

²Abstract of paper submitted to *The Physical Review*.

9.3 ALTERNATING- AND DIRECT-CURRENT CONDUCTIVITY OF GLASSES

R. A. Weeks

E. Lell³

Insulators which have been electrostatically charged have been observed to discharge, leaving in the insulator visible tracks along which the discharge took place.⁴ The electrostatic charging can be induced on the surface or in the bulk of an insulator by electron or gamma-ray irradiation.^{5,6} Since these discharges have been observed in some glasses and not in others, there is some interest in the process by which charge and energy, supplied during irradiation, were stored in such insulators. Glasses with a high ($\sim 10^{19}$ ohm-cm) and some with a low ($\sim 10^{17}$ ohm-cm) intrinsic resistivity exhibit the discharge effect when irradiated with electrons. Since the discharge effect involves electrical processes, it seemed reasonable to investigate the electrical properties of the glasses of interest. The available data^{4,7,8} on the effect indicated that the decay time for its disappearance was quite long, and thus our interest was directed to a study of the low-frequency and dc conductivity of several glasses.

In Fig. 9.1 the loss tangent (ac conductivity) of a lead silicate glass is shown as a function of frequency and time after electron irradiation. There are two features of these measurements that should be noted: (1) there is a maximum in the loss tangent at some frequency below 50 cps, and (2) at least the high-frequency tail of this maximum disappears in a time of the order of 24 hr. It has also been observed that two glasses, exhibiting the discharge effect, also show the greatest increase in the loss tangent at 50 cps. Since this increase disappears in ~ 24 hr, and the discharge effect can be observed after a time >200 hr, the two phenomena are probably not directly related.

³Guest scientist from Bausch and Lomb, Inc., Rochester, N. Y.

⁴S. Whitehead, *Dielectric Breakdown of Solids*, p 170, Oxford University Press, London, 1951.

⁵B. Gross and P. V. Murphy, *Nukleonik* 2, 279 (1960).

⁶Cover picture of *J. Appl. Phys.* 32 (1961).

⁷V. Cullen, in *Proc. Hot Lab. Equip. Conf.*, 7th, Cleveland, 1959.

⁸R. C. Hudson and R. A. Weeks, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, p 89.

The dc conductivity was measured in the lead silicate glass and in a high-purity silica. Within the range of specimen thickness and applied voltages, the observed final current was ohmic. The electrodes were silver paint.

An analysis of the decay of the current through the specimen, the polarization current, showed that it could be reasonably fitted to a function of the form

$$i = \sum_{i=1}^K a_i e^{-t/\tau_i} + bi_o,$$

where a_i is a constant appropriate to the characteristic time τ_i , b is a constant for a particular glass, and i_o is the current observed after a time long compared with the longest τ_i and is presumed to be the intrinsic conductivity. In Fig. 9.2 a decay curve is shown of a lead silicate glass which had been given a Co^{60} gamma-ray dose of 10^6 r. An analysis of the curve gave four characteristic times. The numbers in parentheses are the values of the a_i 's which are the intercepts at $t = 0$.

In Table 9.1 the τ_i 's found by this analysis are shown for two specimens subjected to gamma-ray irradiation and measured at about 25°C and at two higher temperatures. The average values of the

Fig. 9.1. Loss Tangent of a Lead Silicate Glass at Various Times After an Electron Irradiation with $1.5 \mu\text{a}$ of 1.5-Mev Electrons for 5 min. The temperature during irradiation was $\sim 30^\circ\text{C}$ and during measurement was 23°C .

Fig. 9.2. Polarization Current Resolved into Four Processes, Each with a Characteristic Time. The specimen had been exposed to 10^6 r of Co^{60} gamma rays prior to the measurement. (The constant term bi_0 was first subtracted, leaving the linear portion labeled as τ_4 , which was then subtracted from the remainder, giving the linear portion labeled τ_3 , etc.)

τ_i 's are given for the unirradiated and irradiated conditions and the rms deviation calculated. At the elevated temperatures the τ_i 's exhibit a decrease that is larger than this rms deviation.

Although the τ_i 's showed only small changes with these treatments, the a_i 's and the intrinsic conductivity bi_0 exhibited larger variations. These are shown in Table 9.2. Up to a dose of 10^7 r, the a_i 's and the bi_0 increased monotonically with increasing dose with the exception of a_1 . The effect of elevated temperatures was to increase the a_i 's with the exception again of a_1 , for which there was a large decrease.

Discussion

Taylor has shown⁹ that the increase in the loss tangent at low frequencies (Fig. 9.2, curve for the

unexposed case) is due to a distribution of relaxation times for sodium ion diffusion. The increase that we observed may have been due to the same mechanism. The increase in the loss tangent resulting from irradiation could be caused by a change in this distribution. However, Taylor's results showed that there was no change in the distribution with heating, and our τ_i 's derived from the dc measurements showed very little, if any, change with irradiation. It would be questionable on the basis of these results to relate the increase in loss tangent to a change in the distribution of relaxation times for ion diffusion. It is possible that irradiation displaces ions from deep traps and that while they are removed from the traps they can

⁹H. Taylor, *Trans. Faraday Soc.* 52, 873 (1956).

Table 9.1. The DC Polarization Relaxation Times of a Lead Silicate Glass

Treatment	τ_1	τ_2	τ_3	τ_4
Specimen III (unexposed)	3.1	13.8	62	366
One week		14.0 ^a	62 ^a	334 ^a
Turned ^b	3.1	14.5	62	336
10^5 r Co ⁶⁰	2.5	14.0	65	360
2×10^5 r	2.8	13.8	68	375
5×10^5 r	3.0	17.0	66	380
1×10^6 r	3.0	15.0	65.5	375
Specimen IV				
5×10^5 r Co ⁶⁰	3.0	16.5	64	395
1×10^6 r	3.0	16.0	62.5	390
Average value	2.94 ± 0.20^c	15.0 ± 1.4^c	64.2 ± 2.0^c	368 ± 20^c
Specimen IV (unexposed)				
43°C	2.3	12.2	57.5	345
68°C	2.2	12.2	51.0	330

^aSpecimen III was removed from the specimen mount and stored in a dry atmosphere for one week. The recorder was not turned on soon enough to record τ_1 .

^bSpecimen III was removed and then reinserted in about 10 min, reversed with respect to the applied voltage.

^cThe error is the rms deviation from the average value.

contribute to the ion diffusion and are observed as an increase in the loss tangent. Over a period of time these ions would be retrapped, thereby producing the observed decay in the irradiation-enhanced loss tangent. Since the irradiation can also generate electrons and holes, these can alter the ionization states of such traps, and a fraction of the ions released by the irradiation would not be retrapped. These ions would then produce the enhancement of the intrinsic conductivity observed (Table 9.2).

The hypothesis that the time-dependent decay of the current through the specimen can be expressed as a sum of exponentials in time is based on the following facts:

1. The principle of superposition is obeyed. The observed decay curves can be resolved into linear sections which extend over two decades of current. The τ_i 's for several measurements under a variety of conditions have an rms deviation that is less than $\pm 9\%$ for τ_2 and less

than $\pm 6\%$ for the other three τ 's. These rms deviations indicate a reasonable agreement between the hypothesis of superposition and the observed curve. Other functions have not been analyzed, and it is possible that with four adjustable parameters a different function could be found which would fit the observed curve.

2. Ohm's law is applicable for the conditions of our experiment.

3. The final conductivity over a limited range of temperature obeys an Arrhenius equation but deviates at higher temperatures. This deviation could result from two independent processes.

4. The polarization currents found in other experiments on glasses^{9,10} and in other materials^{11,12} exhibit an exponential time depend-

¹⁰R. J. Charles, *J. Appl. Phys.* **32**, 1115 (1961).

¹¹R. W. Dreyfus, *Phys. Rev.* **121**, 1675 (1961).

¹²J. R. Freeman *et al.*, *Rev. Mod. Phys.* **33**, 553 (1961).

Table 9.2. Values^a of the Intercepts of Each of the Linear Portions of the DC Polarization Curves of a Lead Silicate Glass

Treatment	τ_1	τ_2	τ_3	τ_4	bi_0
Specimen III	260	51	25	5.8	1.6
One week		61 ^b	18.8 ^b	5.6 ^b	1.4 ^b
Turned ^c	270	109	30.3	11.0	2.0
10^5 r Co ⁶⁰	22	195	111	40	5.0
2×10^5 r	220	253	123	38	7.0
5×10^5 r	450	345	210	59	8.0
1×10^6 r	315	355	230	74	10.0
Specimen IV ^d					1.4
5×10^5 r	570	420	220	53	11.0
1×10^6 r	420	405	255	70	10.0
Specimen II					
1×10^7 r	750	880	410	180	30
1×10^8 r	2500	500	265	175	49
Specimen III					
43°	390	150	45	14	3.5
68°	92	250	61	25	10.0

^aThe numbers given in the table are the voltages in millivolts measured across the series resistor of the vibrating reed electrometer. Its value was 10^{12} ohms.

^bSpecimen III was removed from the specimen mount and was stored in a dry atmosphere for one week. The recorder was not turned on soon enough to record τ_1 .

^cSpecimen III was removed and then reinserted in about 10 min, reversed with respect to the applied voltage.

^dThe chart recorder failed during the course of the measurement, and only the final conductivity was measured.

ence. The fundamental assumption in these experiments is that there is a periodic array of potential wells in which charge carriers are trapped. In the absence of a field, these charge carriers and the associated charges of the structure creating the potential wells have a net dipole moment of zero. The application of an electric field changes the relative depth of the wells along the field direction, and these are preferentially filled by charge-carrier diffusion. The intrinsic conductivity of the glasses is due to such a process.^{9,11,13} Another process

closely related to this one and which exhibits an exponential dependence on time, is the reorientation of dipoles already present in the system. In this case neither charge component of the dipole diffuses away from the other, and there are several orientations possible, with each separated by a potential barrier. The τ 's that we have found may be the relaxation times of such a process.

One apparent contradiction between the experimental results and this hypothesis is that a thermal activation energy of ~ 1 eV should be observed. In the temperature range over which measurements have been made (Table 9.1), the τ 's appear to be temperature independent as

¹³R. J. Maurer, *Phys. Rev.* **59**, 691 (1941).

compared with the intrinsic conductivity. The polarization process characterized by the τ 's may not be thermally activated in the temperature range which we have investigated.

Summary

The data do not support a space-charge hypothesis. There is agreement with the hypothesis

that the polarization effects occur in the bulk of the glass.

The bulk polarization processes which have long, approximately temperature-independent relaxation times may furnish a mechanism by which charge and energy are stored. There does appear to be a correlation between large values of the τ 's and the discharge effect.

10. Superconductivity

10.1 DEPENDENCE OF SUPERCONDUCTING CRITICAL CURRENT OF Nb-Zr ALLOYS ON MAGNETIC-FIELD ORIENTATION

S. T. Sekula R. W. Boom¹
C. J. Bergeron²

There is a growing class of superconducting materials which exhibits zero resistance in magnetic fields much higher than the thermodynamic critical magnetic field. Such behavior is attributed to a "sponge" of fine filaments whose diameter is of the same order or less than the superconducting penetration depth ($\sim 10^{-6}$ cm).³ These high-field filaments are variously associated with dislocations, highly strained regions, impurities, and other extended imperfections. This indeed is borne out to some extent by the observation that cold working enhances the critical current at high fields.

In a study of the critical current of cold-worked Nb-Zr alloys of varying composition in a magnetic field,⁴ we have investigated the materials in both externally applied transverse and longitudinal fields in order to gain some insight as to the properties of these threads. Figure 10.1 shows data obtained on Nb-33% Zr wires in a cold-worked state and after a heat treatment in a hard vacuum, and is characteristic of a series of Nb-Zr alloys. The critical-current curve of the cold-worked material in the transverse configuration agrees quite well with other published data. The critical current of the longitudinal sample was

observed to increase with increasing field, and a maximum was found at 30 kilogauss. Heat treatment of this material at 800°C increased the critical current at low fields by a considerable amount in both configurations. In addition, the shape of the critical-current curves was greatly altered. X-ray analysis of cold-worked and heat-treated material revealed no change in the preferred orientation of the wire. Metallographic

Fig. 10.1. Orientation Dependence of the Critical Current of Nb-33% Zr Wires in a Magnetic Field. Data are shown for cold-worked and heat-treated material.

¹Electronuclear Division.

²Consultant to Electronuclear Division from Louisiana State University, Baton Rouge.

³J. E. Kunzler, *J. Appl. Phys.* 33, 1042 (1962).

⁴Electronuclear Div. Ann. Progr. Rept. Dec. 31, 1961, ORNL-3257, p 46.

examination⁵ of the cold-worked material showed that appreciable macro- and microsegregation existed across a diameter and along a longitudinal section of the wire. Similar examination of the material heat-treated at 800°C for 30 min indicated that the microstructures were quite similar to those of the cold-worked state, with the addition of a considerable amount of a precipitate, too fine to resolve optically, of unknown structure and composition. By inference from other studies of alloys of similar composition and structure, the heat treatment also resulted in a softening of the wire, indicating that recovery had occurred. There was no evidence that the material had recrystallized.

Similar peaking effects have been observed in highly oriented cold-rolled sheets of Nb-12% Zr (ref 6) and Nb (ref 7) examined in a transverse field. It is interesting to note that critical-current dependence on magnetic field determined experimentally is not in agreement with either the London or Pippard model of current-carrying filaments in magnetic fields.⁸

10.2 SUPERCONDUCTING TRANSITION TEMPERATURE OF PURIFIED TECHNETIUM⁹

M. L. Picklesimer¹⁰

S. T. Sekula

The superconducting transition temperature of technetium has been reported as 11.2°K for powder¹¹ of greater than 99.9% purity and as 9.3°K for arc-melted material¹² made from metal chips contaminated with oxide.

We found that hydrogen-reduced technetium powder presently available contains appreciable quantities of oxygen, both dissolved and as an

unreduced oxide. Microstructural examination of powder particles revealed that each particle was porous, like a sponge, with the dissolved and/or entrapped and unreduced oxide existing as a center between two surface layers of reduced metal.

Five-gram pressed powder pellets were induction-melted under a vacuum of 1 to 5×10^{-6} torr several times until no further evolution of vapor was detected. The resulting buttons were cold-rolled to sheet approximately $\frac{1}{16}$ in. thick. Spectrographic analysis of this material indicated a purity of greater than 99.995% for metallic elements, the only impurity detected being 40 ppm Na. The oxygen content was not determined.

A specimen approximately $\frac{1}{16} \times \frac{1}{16} \times \frac{5}{8}$ in. was cut from the cold-rolled sheet, four platinum leads were spot-welded on, and the specimen and a portion of the leads were encased in cellulose acetate by dipping. The superconducting transition temperature in zero magnetic field was found to be $8.22 \pm 0.01^\circ\text{K}$ by a dc resistance measurement using a current of 0.100 amp and a voltage sensitivity of 1×10^{-8} v.

The decreasing transition temperature of technetium with apparently increasing purity can be rationalized if it is postulated that dissolved oxygen raises the transition temperature. Thus it is likely that the superconducting transition temperature for pure technetium is not greater than 8.22°K. Assurance that the correct value is not lower will have to await the development of analytical techniques for determining the oxygen content of technetium.

10.3 MECHANICAL PROPERTIES OF Nb-25% Zr WIRE AT LOW TEMPERATURES

S. T. Sekula

J. K. Redman

Since the use of superconductors in solenoids is feasible for the generation of high magnetic fields, it is understandably necessary to know the tensile properties of these materials at low temperatures. In any serious design of a solenoid, the stresses that arise due to the interaction of magnetic field and current must be considered. The material investigated was 0.010-in.-diam Nb-25% Zr wire, commercially made by the Wah Chang Corporation, and given to us by R. W. Boom and L. D. Roberts.

⁵Work done by M. L. Picklesimer, Metals and Ceramics Division.

⁶Hake, Berlincourt, and Leslie, *IBM. J. Res. Develop.* **6**, 119 (1962).

⁷Leblanc and Little, p 362 in *Proceedings of the 7th International Conference on Low Temperature Physics*, University of Toronto Press, 1960.

⁸J. G. Daunt *et al.*, *Cryogenics* **2**, 212 (1962).

⁹Submitted for publication in *Physical Review Letters*.

¹⁰Metals and Ceramics Division.

¹¹J. G. Daunt and J. W. Cobble, *Phys. Rev.* **92**, 507 (1953).

¹²V. B. Compton *et al.*, *Phys. Rev.* **123**, 1567 (1961).

Tensile tests were made on an Instron testing machine which applies a constant strain rate to the sample with simultaneous recording of the tension on the sample. Modified pin vises were used to grip the wires. Provisions were made to immerse the samples in either liquid nitrogen or liquid helium during the test. At a given test temperature, samples of various lengths were examined in order to eliminate errors in the modulus of elasticity due to strains in the system other than the sample.

The results at three temperatures are shown in Fig. 10.2. Since the statistics are meager, no significance as yet can be given to the decrease in the modulus in going from room temperature to liquid-nitrogen temperature. At room temperature the load at fracture was found to be 20.6 ± 0.5 lb, while in liquid nitrogen the load was 25.7 ± 1.0 lb. At liquid-helium temperature the scatter in the load at fracture was even greater, the value in

this case being 29.0 ± 2.0 lb. The moduli of elasticity for the three temperatures are given in the figure.

10.4 NEUTRON IRRADIATION OF SUPERCONDUCTING TIN

S. T. Sekula

C. E. Klabunde

Studies of the effect of radiation damage in superconductors at low temperatures (4°K) have been limited to observations of the change in superconducting transition temperatures.¹³ Blanc *et al.*¹⁴ recently studied the magnetization curves

¹³Rinderer and Schmid, p 395 in *Proceedings of the 7th International Conference on Low Temperature Physics*, University of Toronto, 1960.

¹⁴Blanc *et al.*, p 393 in *Proceedings of the 7th International Conference on Low Temperature Physics*, University of Toronto, 1960.

Fig. 10.2. Stress-Strain Curves of 0.010-in.-diam Nb-25% Zr Wires at 300, 77, and 4.2°K .

of tin and rhenium subsequent to neutron irradiation at 77°K. However, since radiation-induced defects are quite mobile at 77°K, it is desirable to irradiate and to examine the materials at low temperature and to anneal the material in a controllable fashion. With the introduction of point defects by low-temperature irradiation, one might expect a change in the superconducting transition temperature.¹³ Annealing at higher temperatures may result in a precipitation of point defects as extended imperfections, for example, dislocation loops, which would then be manifest in either a hysteresis in the magnetic transition¹⁴ or, as Shaw and Mapother¹⁵ have demonstrated in cold-worked materials, a broadening of the superconducting-to-normal resistive transition.

As a preliminary experiment the superconducting-to-normal resistive transition of a pure tin wire as a function of current passing through the wire was examined at several temperatures. Figure 10.3 shows the results obtained at 3.60°K. These results are in good agreement with the London model of the intermediate state, which predicts

$$\frac{R}{R_n} = \frac{1}{2} \left(1 + \sqrt{1 - \frac{(I_c)^2}{I^2}} \right), \quad I \geq I_c,$$

where R_n is the normal residual resistance, and I_c is related to the radius of the wire r and the thermodynamic critical field H_c at a given temperature by the relation $H_c = 2I_c/r$. The superconducting-to-normal transition is characterized by a sharp rise in resistance at $I = I_c$ and an asymptotic increase with further increments of current.

A similar sample of tin was placed in the Hole 12 low-temperature facility for an exposure of one week, corresponding to a neutron dose of 5×10^{17} neutrons/cm². Examination of the resistive transition in the reactor prior to irradiation revealed that the transition was smeared

Fig. 10.3. Superconducting-to-Normal Transition of Tin at 3.60°K.

out. This is due to small uncontrollable temperature gradients caused by local gamma-ray heating. In addition, the vapor pressure thermometry was found to be inadequate for observing the small changes in the transition temperature one would expect after irradiation. No change was observed in the resistive transition after the irradiation or after subsequent 3-min anneals at 25, 70, and 300°K. Thus, within the limitations of the techniques, there was no effect of neutron irradiation on the superconducting transition of tin.

Blanc *et al.*¹⁴ report a small decrease in the transition temperature and a small increase in the frozen-in moment in a tin sample after a neutron dose of 10^{18} neutrons/cm² at 77°K. On the other hand, Shaw and Mapother¹⁵ found no significant change in the magnetic transition of tin severely cold worked at 4.2°K. We hope to improve the techniques and extend the study to other materials.

¹⁵Shaw and Mapother, *Phys. Rev.* 118, 1474 (1960).

10.5 INTERACTION BETWEEN IRRADIATION-INDUCED DEFECTS AND MAGNETIC STRUCTURE¹⁶

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R. H. Kemohan

P. G. Huray¹⁸

W. L. Harman¹⁹

Hysteresis loop measurements have been conducted on several commercial iron-nickel alloys

¹⁶To be submitted for publication in "Crystal Lattice Defect Supplement," *Journal of the Physical Society of Japan*.

¹⁷U. S. Naval Research Laboratory, Washington, D. C.

¹⁸Co-op student from the University of Tennessee, Knoxville.

¹⁹Co-op student from the Virginia Polytechnic Institute, Blacksburg.

during neutron irradiations at 90°K and at ambient reactor temperature in both zero and saturating magnetic field. Isochronal annealing tests were also performed on the alloys after irradiation. The low-temperature irradiation results indicated that magnetic properties are affected not by the point defects themselves but by their motion and eventual disposition. The radiation-produced changes appeared similar to those observed in these alloys upon magnetic annealing, that is, short-range atomic ordering. Further irradiation experiments on the alloys at temperatures from 70 to 175°C in a saturating magnetic field showed that the activation energy of the radiation-induced process was 0.3 ev. However, in all cases the radiation-induced changes were annealed out at temperatures below the usual ordering temperature. This recovery process appeared to be dependent upon the state of magnetization.

Part III. Metals

F. W. Young, Jr.

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11. Investigations of Metal Surfaces

F. W. Young, Jr.

U. Bertocci

L. D. Hulett

L. H. Jenkins

J. R. Savage

Electrochemical techniques offer a reasonable method of studying the reactivity of metal surfaces, since experimental conditions can be so adjusted that data can be obtained from systems in which the magnitude of either the current flowing or the potential measured at the electrode can be controlled. The electrical parameters observed for electrodes can, under certain conditions, be related to resultant changes in electrode surface structures, which are observed by optical and electron microscopy. While both crystal growth and dissolution may be examined profitably by such techniques, the emphasis in this work has been on the study of anodic processes under conditions of constant current.

Two possible conditions for the study of anodic processes are: (1) dissolution into solutions containing ions of the electrode metal, and (2) dissolution into solutions which initially contain few, if any, ions of the metal. Near equilibrium conditions, electrode potentials measured under condition (1) may be correlated sometimes with the energy required to remove material from sites related to the crystallographic orientation and/or the defect structure of the electrode surface. In case (2) the definition of an equilibrium condition with respect to oxidation of the metal is difficult and is of little practical interest. Also, concentration gradients at the electrode-solution interface play a predominant role in determining the emf values measured and the distribution of current densities over the metal surface. Since it is possible that dissolution mechanisms operative at the electrode surface may be affected by changes in such concentration gradients, systems of this type are also of interest.

11.1 ANODIC DISSOLUTION OF COPPER IN SOLUTIONS CONTAINING COPPER IONS

Studies of the galvanostatic dissolution of copper single crystals in oxygen-free 0.2 M CuSO_4 (pH adjusted to ~ 1 with H_2SO_4) have been continued. Observations on (100) oriented surfaces have been reported previously,¹ and data are now being gathered on surfaces oriented (110) and (111). The latter orientations exhibit relationships of current vs potential similar to those observed for the (100) surface. However, it has not yet been possible to demonstrate that discontinuities in the curves resulting from a plot of overpotential vs current density are related to the defect structure of the electrode in the case of (110) and (111) orientations, as was shown for (100).

It has been observed that without application of an external emf, (110) and (111) oriented surfaces, unlike the (100), develop a faceted structure within 24 hr after exposure to the copper(II) sulfate solutions. Also, it has been demonstrated that the electrodes do not change weight under such conditions. The faceting must then result from the transfer of material from one area of the electrode to another; and, therefore, anodic and cathodic areas are localized over the electrode surface. It is reasonable to assume that such behavior prevents the correlation between measurable electrical parameters and the surface defect structure similar to that observed for (100) surfaces.

¹L. H. Jenkins and J. O. Stiegler, *J. Electrochem. Soc.* **109**, 467 (1962).

Current studies are directed to determining the conditions which control the development of facets on electrodes at chemical equilibrium, as well as establishing the experimental conditions necessary to demonstrate a relationship between electrical parameters and the defect structure of (110) and (111) surfaces in these solutions.

11.2 ANODIC DISSOLUTION OF COPPER IN COPPER-FREE SOLUTIONS

In an effort to determine the parameters affecting the formation of etch pits at dislocations, anodic dissolution experiments, by means of an external emf, have been carried out mostly on the (111) surface of copper single crystals.

It seems that in every case where etch pits are formed at dislocations, copper is dissolved in its univalent form. Thus, dislocation pits were observed in solutions of HCl, HCl + HBr, HBr, HCl + HI, and $\text{NH}_4\text{OH} + \text{NH}_4\text{Br}$. On the other hand, pits did not develop at dislocations in solutions of HClO_4 , H_2SO_4 , and CH_3COOH , in which copper is anodically oxidized to the divalent state. No clear-cut results have been obtained for KCN or NH_4OH solutions, although copper is oxidized to the univalent state and there is a tendency to form pits which apparently do not correspond to dislocations.

Current Density and Concentration of Solutions

The effect of current density upon dislocation pit formation has been investigated. For HCl and HBr solutions and their mixtures, the lowest current-density limit is set by the experimental method, since at current densities less than 0.1 ma/cm^2 the presence of oxygen in solutions makes the results unreliable. The concentration of components is also an important parameter; in HCl solutions the current-density range within which pitting is observed is rather narrow, but the addition of small quantities of HBr or HI extends this range considerably. A concentration of 0.1 mole of HBr per liter is sufficient to raise the upper limit for pitting from 5 to 80 ma/cm^2 . In the case of HI a concentration of 3×10^{-4} mole/liter allows pitting up to about 30 ma/cm^2 . The presence of copper ions in HCl solutions, previously equilibrated with copper metal, has an adverse

influence on pit formation at dislocations. A concentration of 0.05 mole of copper per liter is enough to prevent pit formation. However, if HBr is added to such solutions, pitting is again observed.

A lower limit of current density at which pits developed has been observed in solutions containing $0.3 \text{ M NH}_4\text{Br}$ in 1.5 to $5 \text{ M NH}_4\text{OH}$. Dislocation pits are formed here only if the current density is more than 30 to 40 ma/cm^2 . This limit seems to shift toward higher values as the NH_4OH concentration is increased.

In HCl solutions an upper limit of 5 to 6 ma/cm^2 has been observed which seems to be independent of the HCl concentration in the range tested, 2 to 6 M . For HBr and HCl-HBr mixtures, where HBr is not less than 0.1 M , the upper limit for pitting at dislocations is affected by stirring, by time, and by the precipitation of salt on the electrode; it is therefore dependent on concentration of the acids, as it affects the solubility of the cuprous salt. Current densities up to 150 ma/cm^2 could be used in the most concentrated solutions. The current density also affects the shape of the pits developed in such solutions. Generally, low current densities produce quasi-hexagonal pits as shown in Fig. 11.1a, while high current densities result in almost perfect triangles like that in Fig. 11.1b.

Orientation Effects

In the HCl-HBr system the effect of bromide concentration and current density on the angular spread of the pitted area about the (111) pole on spherical crystals has been examined. The solutions investigated most thoroughly were 6 M in HCl, with bromide concentrations varying from 0.1 to 0.5 M . In such solutions, when current density increases, a maximum occurs in the angular spread of the pitted area. As bromide ion concentration is increased from 0.1 M to 0.5 M , the maximum in angular spread increases over the range 80 to 110 ma/cm^2 .

For current densities below the maximum, the ratio of the depth of an individual pit to its width likewise increases with current density. Also, the crystallography of the pits is independent of the orientation of the surface. A deviation of the surface from the (111) changes the section cut in the surface by the pyramid bounding the

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(a)

(b)

Fig. 11.1. Effect of Current Density on the Shape of Pits Developed in Acid Halide Solutions. (a) 5 ma/cm². 550X. (b) 60 ma/cm². 400X. Reduced 17%.

pit. This effect is shown in Fig. 11.2, which is a diagram of the pitted area around a (111) pole. The symmetrical triangle in the center of the drawing represents the shape of a pit produced on perfectly oriented (111) surfaces. The distorted triangles represent the typical shapes of pits produced on surfaces slightly misoriented from the (111). A misoriented surface will have observable pits only if the sides of the pits are inclined to the (111) at angles greater than the angle of misorientation. Thus, an increase in current density by increasing the average depth/width ratio of the etch pits will increase the allowable angular spread of the pitted area.

In addition to experiments on the (111), some work has been done on (100) and (110) surfaces.

Fig. 11.2. Geometry of Pits as Affected by Slight Misorientations from the (111) Orientation.

Etch pits at dislocations can be developed by anodic attack on these surfaces also. Dissolution of (100) surfaces in an acid chloride-bromide solution produces etch pits at dislocations. However, the pit pattern has a large amount of random faceting that makes interpretation difficult. Subgrain boundaries are clearly delineated as rows of square pits, but isolated pits are sometimes difficult to distinguish from the surrounding square facets. Subgrain boundaries on the (110) can be displayed by anodic etching at low current densities, approximately 2 ma/cm² in HCl solutions.

11.3 KINETICS OF ETCH-PIT GROWTH AND ASSOCIATED PHENOMENA ON (111) COPPER SURFACES IN ACID HALIDES

It has been determined that etch pits can readily be formed at dislocation-surface intersections on the (111) face of copper by making the crystal the anode in an electrolytic cell, with various halide solutions as the electrolyte. The kinetics of the growth of the etch pits, and associated phenomena on the surface, is being studied by

performing galvanostatic etching experiments under the optical microscope and by using time-lapse photomicrography. An example of the type of information obtainable by this technique is shown in Fig. 11.3, which is a sequence of photomicrographs taken at 10-sec intervals of a (111) copper surface being electrochemically etched at a current density of 20 ma/cm^2 in a 6 M HCl , 0.25 M HBr solution. In the accompanying graph, Fig. 11.4, two dimensions of the etch pit, X and Z (the position of a ledge inside the pit and the position of ledges on the surface), are plotted as a function of time. The center of the pit is used as the locus for all measurements; therefore, only the slopes are significant for the ledges. Measurements are also being made, by the use of interferometric microscopy, of the depth d of the pits as a function of their lateral dimensions and of the height of the ledges. A plot of d/X vs time, for the pits formed under the same conditions as for Fig. 11.3 but in a separate experiment, is shown in Fig. 11.5. The relative depth of the pit decreases sharply during the first 30 sec of etching; for longer times d/X is approximately constant.

The dislocation density in this crystal was $2 \times 10^3/\text{cm}^2$. If the pits are assumed to be symmetrical, $\text{volume/pit} = 1.3 \times 10^{-2} X^3$ after about 30 sec. Thus, while the size of the pit increases linearly with time, $X (\text{cm}) = 5.3 \times 10^{-5} t (\text{sec})$, the volume increases cubically with time, $V (\text{cm}^3) = 1.9 \times 10^{-15} t^3$. From the current density, the rate at which copper is removed is equal to $w = 1.4 \times 10^{-6} \text{ cm}^3 \text{ cm}^{-2} \text{ sec}^{-1}$. Thus, during the course of the etching (Fig. 11.1), the relative amount of copper removed from the pits increases to only $\sim 10\%$ of the total volume removed at 190 sec.

The velocity of the ledges on the surface is dependent on the position on the surface, as shown particularly in this experiment. In region A (see Fig. 11.4) the density of ledges is $3.2 \times 10^3/\text{cm}$ and the velocity of motion is $4.3 \times 10^{-5} \text{ cm/sec}$, while in region B the density is $2.4 \times 10^3/\text{cm}$ and the velocity is $3.6 \times 10^{-5} \text{ cm/sec}$. The flux of ledges, ϕ , in region A is $\phi_A = 1.4 \times 10^{-1}/\text{sec}$, while $\phi_B = 0.9 \times 10^{-1}/\text{sec}$. The difference in these values, though small, is thought to be significant. Whether the difference corresponds to small differences in ledge heights L or to microscopic differences in current density is not resolvable at present. If a uniform current density

Fig. 11.3. Growth of Etch Pit (20 ma/cm^2 ; 6 M HCl , 0.25 M HBr). First photograph after 20 sec; 10-sec intervals between subsequent photographs. 180X. Reduced 14%.

Fig. 11.4. Position vs Time for Several Features from Fig. 11.3.

Fig. 11.5. Pit Size and Pit Depth vs Time. Same conditions as for Fig. 11.3.

is maintained microscopically from ϕ and w , L is calculated to be $\sim 10^{-5}$ cm, which agrees with interferometric measurements. On the other hand, by using the value $L \sim 10^{-5}$ cm and the measured

values of the ledge density to calculate the corresponding misorientation, it can be shown that the ledges must have resulted from steps nucleated on the surface as well as ledges arising from slight misorientations of the surface. It is clear from the photographs that while the density of ledges does not change appreciably with time, the height does increase. And it should be remembered that the relative amount of copper being removed from the pits also increases with time. A possible explanation for these observations is that most of the copper is removed from monatomic steps during the early stages of the etching, that these steps bunch to form the multi-atomic ledges, and subsequent removal occurs primarily at the ledges.

The side of the pit nearest ledges B grows at exactly the same velocity as the ledges B move. Since (from the geometry of a pyramidal pit with straight edges) the velocity of the side $\equiv \frac{1}{2} v_X$, the fact that for this experiment the velocity of the side $> \frac{1}{2} v_X$ is just a measure of the bowing out of this side. In a similar way the bowing in of the other sides is apparent and explicable. Since the ledges are moving in the Z direction, it is apparent that Z should be $< X$.

The velocity of a ledge inside the pit is the same as v_X in this experiment. Since the surface is retreating (via ledge and step motion) as the pit grows, it would be expected that the leading ledge of the pit would periodically be removed, and thus v_X would be less than the velocity of a ledge inside the pit. It would seem that the only way these two velocities could be equal is that the distance between the ledges in the pit increases near the edge of the pit. That this is true is apparent both from examination of the photomicrographs and from interferometric measurements.

One of the important observations is that the dislocations are not the sources of the ledges on the surface; evidently they arise both from surface nucleation and from slight misorientations of the crystal surface.

The kinetics of pit growth and of ledge motion are being studied in the manner described above as a function of the variables current density, concentrations of Cl^- and Br^- , dislocation density, and orientation.

11.4 GROWTH OF COPPER CRYSTALS WITH LOW DISLOCATION DENSITY

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A study is in progress to determine the factors controlling dislocation density in copper crystals grown from the melt. Copper of 99.999% purity (supplied by ASAR Company) is used as the stock material and is carefully cleansed by etching so as to avoid the introduction of impurities. The crystals, 1 in. in diameter by 6 in., are grown in a graphite (chlorinated C-18, National Carbon Company) crucible by use of a Bridgman technique at a rate of 1 in./hr. Approximately 125 nonseeded crystals have been grown, and for apparently identical conditions of growth the orientations of the growth direction were scattered over the stereographic triangle. The crystals were sliced to obtain (111) or (100) faces, via acid sawing and acid polishing,² and the dislocation densities throughout the crystals were determined by etch-pit techniques. A definite correlation between growth direction and crystal perfection was observed,³ the dislocation density being an inverse function of the angular deviation of the growth direction from the (111) zone. Crystals with a dislocation density of $\sim 10^3/\text{cm}^2$ and with no subboundaries were grown.

Attempts to grow seeded crystals, (111) growth direction, with low dislocation density have been unsuccessful to date. Apparently, during the seeding process the dislocation density is greatly increased; and although the growth direction is good, the dislocations are propagated during growth into the new crystal. These studies are being continued.

It has been found that subsequent annealing of the crystal slices for one week at 1075°C serves to reduce the dislocation density, and a number of samples with a dislocation density of $10^2/\text{cm}^2$ have been obtained (see Fig. 11.6). The mechanism of this annealing process is not clear, but the dislocation density has been reduced from 5×10^4 to $\sim 5 \times 10^2/\text{cm}^2$ on a reasonably routine basis.

In order to determine the dislocation densities in copper by independent methods, specimens of low dislocation density are being prepared for transmission x-ray and electron microscopy. Thin

Fig. 11.6. Etch Pits at Dislocations on Copper Crystals. 3X. (a) As grown. (b) After annealing 1 week at 1075°C . Reduced 24%.

plates are cut with the acid saw, acid polished, and finally thinned by electropolishing. Since copper is so soft, it is very difficult to avoid accidental deformation of these plates, but preliminary results⁴ suggest that specimens suitable for x-ray microscopy can be prepared.

²F. W. Young, Jr., and T. R. Wilson, *Rev. Sci. Instr.* **32**, 559 (1961).

³F. W. Young, Jr., *Bull. Am. Phys. Soc.* **7**, 215 (1962).

⁴M. C. Wittels, F. A. Sherrill, and F. W. Young, Jr., "X-Ray Observations of Nearly Perfect Copper Single Crystals," see abstract this report.

11.5 ETCH-PIT STUDIES OF DISLOCATIONS IN COPPER CRYSTALS DEFORMED BY BENDING: I. ANNEALED CRYSTALS; II. IRRADIATED CRYSTALS⁵

Annealed and irradiated copper crystals of low initial dislocation density were deformed by bending, and dislocation multiplication and motion were studied by use of an etch-pit technique for observing the dislocations. For the annealed crystals there was dislocation motion and multiplication prior to macroscopic yielding, and the percentage of grown-in dislocations moved by the stress increased with the stress. The dislocations were hindered in the motion by subboundaries and by interaction with other dislocations. Relaxation

effects were observed and were much greater for stresses less than the yield stress than for higher stresses. For the neutron-irradiated crystals there was no motion or multiplication of dislocations prior to yielding. The slip traces were long, with dislocations closely spaced in the trace. Although not conclusive, the evidence apparently was best interpreted in terms of source hardening. A cube-root dependence of the yield stress on neutron dose was observed. No hardening resulted from irradiation with $10^{18}/\text{cm}^2$ cobalt gamma rays.

⁵Abstract of paper submitted to *Journal of Applied Physics*.

12. Low-Temperature Irradiation Studies

R. R. Coltman

C. E. Klabunde

D. L. McDonald¹

J. K. Redman

12.1 REACTOR DAMAGE IN PURE METALS AT 4°K

By relocating the fuel in the vicinity of a liquid-helium cryostat located in the ORNL Graphite Reactor, it has been possible to separate the thermal and fast components of the reactor neutron flux. Studies of the radiation damage effects arising from each type of flux have been made. It has been found that an appreciable fraction of the reactor damage in several metals arises from thermal neutrons. The effect results from the recoil of an atom from the (n,γ) reaction at the time of thermal-neutron capture. The low-temperature recovery of thermal-neutron damage is greater and shows more annealing peak structure than the recovery of fast-neutron damage. Thermal-neutron damage concentration studies have been made on cadmium, and pronounced suppression of the annealing is found as the concentration is increased. The mean primary recoil energy from a thermal-neutron capture event has been calculated for several elements. Values range from about 50 ev for the heavier elements to several hundred electron volts for the lighter elements. The relative effects of atomic recoils from decay events and decay beta-atom collisions are estimated and found to be small compared with (n,γ) recoil effects. Damage from fission neutrons has been studied in copper, and it was found that the recovery of this damage is suppressed compared with that obtained from reactor fast neutrons.

The summary above describes the major portion of the work performed by the group during the past year. A detailed report of this work has been accepted for publication in the *Journal of Applied Physics*.

12.2 INTENSE DAMAGE IN CADMIUM

In the temperature range 4 to 40°K the isochronal annealing behavior of thermal-neutron-irradiated cadmium shows a well-behaved suppression of the recovery as a function of neutron dose. Changes in residual resistivity were used to observe this effect. The mean recoil energy of a cadmium atom at the time of thermal-neutron capture (n,γ reaction) is calculated to be 130 ev. Although the threshold displacement energy for cadmium is not known, one may use similar results for copper, silver, and nickel to estimate that each capture event in cadmium might make between one and two Frenkel pairs. Thermal-neutron damage in cadmium, then, should be uniformly distributed as contrasted with fast-neutron damage, which is believed to occur in localized regions with a high damage concentration. The isochronal recovery of fast-neutron damage in cadmium is considerably suppressed compared with the recovery from various modest doses of thermal-neutron damage. It is believed that if the thermal-neutron damage concentration could be increased to the point where its recovery resembled that of fast-neutron damage in the temperature range 4 to 40°K, then one might make a rough estimate of the defect concentration resulting from a typical fast-neutron hit. To achieve such a high concentration, the proper amount of Cd^{113} isotope was added to natural cadmium to produce a specimen

¹Guest scientist from the Australian Atomic Energy Commission.

with twice the capture cross section of natural cadmium. The specimen was then bombarded at 4.2°K for two weeks to an integrated thermal-neutron flux of $6.5 \times 10^{17} \text{ nvt}$. The average concentration of capture events in this specimen was 0.2 at. %, which might produce between 0.2 and 0.4 at. % of Frenkel pairs. In Fig. 12.1 we see the isochronal recovery of this specimen along with that obtained after a substantially pure fast-neutron bombardment. (The recovery from a thermal-neutron dose one-half as great is also shown to illustrate the suppression effect due to dose.) It can be seen that the recovery from this high thermal-neutron dose closely approached that obtained from a fast-neutron irradiation. From this it is estimated that the defect concentration in a damaged region resulting from a fast-neutron hit is between 0.3 and 1.0 at. % Frenkel pairs.

12.3 NEW LOW-TEMPERATURE IRRADIATION FACILITY FOR THE BULK SHIELDING REACTOR

From the work completed this year it is clear that thermal neutrons make a significant contribution to the total reactor flux damage in many elements. The study of pure thermal-neutron damage is of particular basic interest for a large number of elements, since the primary atom recoil energies are relatively small. For the present work, thermal-neutron damage studies have been made by retracting the fuel in the vicinity of the liquid-helium cryostat located in the ORNL Graphite Reactor. This causes a substantial reduction in the fast-neutron flux, while the thermal-neutron flux remains unchanged. It is apparent, however, that for the detailed study of thermal-neutron

Fig. 12.1. Isochronal Recovery of Irradiated Cadmium.

damage in many elements, a further reduction of the fast-neutron flux is necessary. The present fuel configuration around the cryostat (a 44-in.-diam fuelless sphere) is the terminal fuel retraction that can be made. Any additional fuel retraction would make the reactor inoperable. To extend the thermal-neutron damage studies and to obtain virgin fission neutrons by the use of unmoderated U^{235} converters, construction of a low-temperature irradiation facility to be associated with the BSR has begun. The initial effort will consist in placing a rectangular tank of heavy water between a face of the reactor and a liquid-helium cryostat. The tank will be 12 in. thick and will cover the entire reactor face.

In order to obtain an idea as to what damage effects might be expected in such a facility, an existing D_2O tank, differing from the one above in that it did not quite cover the entire reactor face, was installed. M. S. Wechsler and J. M. Williams made some preliminary flux determinations adjacent to the tank, and J. W. Cleland and R. F. Bass obtained a solid-state characterization of the fast flux by measuring the removal rate of conduction electrons in n -type germanium. The monitors used in the flux determination were Co^{59} , U^{238} , Ni^{58} , and S^{32} . The techniques are discussed elsewhere in this report (see "Fundamental

Studies," Part V). Table 12.1 shows the values of the various measured fluxes behind the D_2O tank compared with values of the same quantities at the face of the BSR and at two positions in the ORNL Graphite Reactor.

The removal rate of conduction electrons in n -type germanium has been determined as 3.2 per incident fast (damaging) neutron, wherein it has been assumed that the uranium doughnut fission chamber in Hole 51 of the ORNL Graphite Reactor had an interior fast (damaging) flux of $8 \times 10^{11} \text{ cm}^{-2} \text{ sec}^{-1}$. Two n -type germanium samples were irradiated for 1 hr outside the 12-in. tank of D_2O . The power level was 1 Mw. The samples were contained inside a $\frac{3}{8}$ -in. boron carbide impregnated plastic container that has been shown to eliminate any effect from either thermal neutrons or capture gamma rays. The total conduction electron removal was 5.2 and $6.0 \times 10^{14}/\text{cm}^3$ for the two samples. If one assumes a removal rate of 3.2 per incident fast (damaging) neutron, as above, then the total fast (damaging) neutron flux exterior to the tank of D_2O was $4.9 \times 10^{10} \text{ cm}^{-2} \text{ sec}^{-1}$ in this experiment. It is believed that this value is in reasonable agreement with the threshold detectors indicated above, in that the germanium samples are probably sensitive to all neutrons above 1 kev neutron energy.

Table 12.1. Neutron Flux Behind D_2O Tank in BSR Compared with Flux at the Face of BSR and Flux in Two Positions in the Graphite Reactor

Monitor	Measured Flux	Bulk Shielding Reactor		Graphite Reactor, Hole C ^a	
		Face of Reactor	Behind D_2O Tank	Center	Reflector 12 in. from Fuel
Co^{59}	Thermal flux, neutrons $\text{cm}^{-2} \text{ sec}^{-1}$	$1 \times 10^{13}{}^b$	9.1×10^{11}	8.5×10^{11}	4.2×10^{11}
Co^{59}	Ratio, cobalt/cadmium		120	8	21
U^{238}	$\Phi (E > 1.6 \text{ Mev})$, neutrons $\text{cm}^{-2} \text{ sec}^{-1}$	$2.1 \times 10^{12}{}^c$	9.9×10^9	3.4×10^{10}	3.2×10^9
Ni^{58}	$\Phi (E > 2.9 \text{ Mev})$, neutrons $\text{cm}^{-2} \text{ sec}^{-1}$	$9.2 \times 10^{11}{}^c$	5.8×10^9	1.5×10^{10}	1.9×10^9
S^{32}	$\Phi (E > 3.0 \text{ Mev})$, neutrons $\text{cm}^{-2} \text{ sec}^{-1}$	$8.6 \times 10^{11}{}^c$	5.4×10^9	1.3×10^{10}	1.4×10^9

^aM. S. Wechsler *et al.*, "The Effect of Radiation on Atomic Rearrangements in Cu-Al," this report.

^bF. C. Maienschein *et al.*, *Attenuation by Water of Radiations from a Swimming Pool Type Reactor*, ORNL-1891, p 10 (1955).

^cEstimated from neutron spectrum given by F. C. Maienschein in *Third Radiation Effects Symposium* 2, 31 (1958).

The results shown above indicate that the scheme proposed for the BSR will give a higher absolute value of thermal-neutron flux as well as a higher ratio of thermal to fast neutron flux than can be attained in the ORNL Graphite Reactor. The use of a larger heavy-water tank (construction completed), which will completely cover a reactor face, and of a large void space between the cryostat and the D_2O tank should further improve both the thermal flux and the ratio of thermal to fast neutrons.

An addition to the south end of the BSR building which will house the refrigeration equipment is about 50% complete. Helium compressor skids, vacuum jacketing for a new heat exchanger, and some instrumentation and controls have also been completed. The heat capacity of the new refrigeration system is estimated to be about 25 w at about 4.5°K. A new cryostat also under construction will have a sample chamber 1 in. in diameter and 9 in. long.

13. Dislocation Interactions

13.1 EVIDENCE FOR VACANCY CLUSTERING IN DISLOCATIONS IN COPPER

D. O. Thompson

One of the problems in the study of radiation damage in metals is concerned with the interaction of radiation-produced point defects with existent defects, such as dislocations. It has been shown previously that these interactions are sufficient, even at extremely small neutron doses, to effect rather drastic reductions in the internal friction and modulus defect of pure copper crystals.¹ From the fundamental viewpoint, part of this problem may be associated with the interaction between defects after they are trapped by the dislocations. For example, Coulomb and Friedel have discussed the growth of voids around dislocations due to excess vacancy concentrations.² This process would be expected to contribute to source hardening of an irradiated material, since the interaction force between a defect and a dislocation is expected to be proportional to the misfit parameter of the defect. A part of the work this year, therefore, has been spent in an effort to determine whether sensitive internal-friction techniques could be used to learn something of defect-defect interactions in existent dislocations after light neutron bombardments with the aid of the Granato-Lücke theory of dislocation internal friction. Thus, the work to be reported is an attempt at a systematic extension of some preliminary results given earlier in which it was suggested that perhaps the neutron-produced dislocation pinning-

point distribution was temperature dependent for a given concentration of defects.³

All measurements included in this set of experiments were made upon a single crystal of high-purity copper in an internal-friction apparatus described elsewhere [D. O. Thompson and F. M. Glass, *Rev. Sci. Instr.* **29**, 1034-40 (1958)]. Once the sample was placed in the bombardment facility, no handling of it took place until the experiments were over. A beam hole (HB-2) of the ORR served as the bombardment facility. The beam holes are equipped with stopcock-type shutters so that the neutron beam may be stopped independently of the reactor's operation. A series of five bombardments in all were given in this set of experiments. These were chosen to give neutron doses of about 3×10^{10} , 6×10^{10} , 12×10^{10} , 24×10^{10} , and 48×10^{10} *nvt*. Between each of these bombardments the neutron beam was extinguished, the sample was aged at 100°C for at least 24 hr so as to stabilize the crystal against any further thermal migration of radiation-produced defects to dislocations, and then the modulus defect and the amplitude-independent and amplitude-dependent internal friction were measured from about 200°K up to the postbombardment aging temperature.

The results of measurements of the amplitude-independent decrement taken at a strain of about 5×10^{-8} as a function of temperature after the first three bombardments are given in Fig. 13.1. Also shown in the upper portion of the figure is the temperature dependence of the preirradiation decrement taken at the same strain amplitude. From this figure it is apparent that successive amounts of irradiation lower the overall values of the decrement and, further, that the temperature dependence of the decrement is altered.

¹D. O. Thompson and D. K. Holmes, *J. Appl. Phys.* **27**, 713 (1956).

²P. Coulomb and J. Friedel, "On the Formation of Cavities Along Dislocations," p. 555 in *Dislocations and Mechanical Properties of Crystals* (ed. by J. C. Fisher et al.), Wiley, New York, 1956.

³D. O. Thompson, *Acta Met.* **10**, 327 (1962).

Fig. 13.1. Temperature Dependence of Small-Amplitude Decrement Before Irradiation and After the First Three Bombardments.

An example of the dependence of the decrement upon the strain amplitude over this temperature range is given in Fig. 13.2. These data were obtained after B-3, the third bombardment in the sequence. The interesting feature of these curves is that they cross at higher strains as the temperature is changed. Thus, with increasing temperature the small strain decrement decreases, but the strain range available before the decrement increases sharply is shortened.

Fig. 13.2. Logarithmic Decrement vs Strain Amplitude After the Third Bombardment at Various Temperatures.

The behavior of the decrement in this temperature range changed as a function of further dose. In Fig. 13.3 is shown a temperature cycle of the small-amplitude decrement after bombardment B-5, the largest dose in this set of experiments. From a low value of about 1×10^{-4} near 330°K following the bombardment, the decrement increased upon cooling as shown in the figure by the open circles. Both time and temperature effects were apparent in the process. Upon warming, the decrement followed the upper path given by the solid circles. A series of 20-hr isothermal anneals in the 360 to 400°K range was sufficient to restore the decrement to the 1×10^{-4} value measured before the low-temperature traverse. An interesting feature to note is that the temperature dependence of the decrement during the warming portion of the cycle is again approximately linear, as in the preirradiated state.

Fig. 13.3. Temperature Cycle of Small-Amplitude Decrement After the Fifth Bombardment. Values are given at the beginning and end of the isothermal anneal.

A change in the decrement-strain amplitude curves accompanies the type of transition shown in Fig. 13.3. In Fig. 13.4 are shown two sets of

Fig. 13.4. Logarithmic Decrement vs Strain Amplitude After the Fourth Bombardment at Various Temperatures.

data obtained after B-4, the fourth bombardment. The lower set of curves was obtained in the first set of measurements following the bombardment. After these were obtained, the sample was allowed to remain at room temperature for several days and was remeasured in the low-temperature range with no intervening high-temperature cycle and with the addition of no more irradiation. The lower set is like those obtained after the first three bombardments, while the upper set is like that obtained after B-5. It will be seen that they are indeed quite different sets of curves: (1) The upper set shows the "normal" temperature dependence, while the lower, or first, set is "inverted." (2) No crossing of the curves occurs in the upper set as it does in the lower. (3) The decrement reaches a plateau as a function of strain in the upper set, which is not the case in the lower set.

An interpretation of these results has been made in terms of the Granato-Lücke theory of internal friction due to dislocations.⁴ The basic experimental results indicate, in terms of this theory, that dislocation pinning points are lost as the temperature is lowered, and are recovered by suitable annealing treatments as it is raised. The recovery is true for all bombardments made

⁴A. Granato and K. Lücke, *J. Appl. Phys.* 27, 583 (1956).

to date, but the recovery temperatures are higher after B-5 than after the early bombardments. Even though it is clear that the simple Granato-Lücke theory is not adequate to explain completely the results, but should probably be amplified to include the effects of at least two kinds of dislocations upon the internal friction and modulus defect, it has been used to calculate the number of pinning points as a function of temperature for constant amounts of irradiation. This has been done with both the amplitude-independent decrement results in Fig. 13.1 and the modulus defect results. The results of such calculations are given in Fig. 13.5 in terms of normalized curves for bombardments 1 and 2. The parameters $\gamma^{-1/2}-1$ and $Z^{-1/4}-1$ are, theoretically, equal to the ratio of the dislocation pinning points added by irradiation to those present before irradiation,⁵ and f is the fraction of pinning points at temperature T divided by the number observed at the temperature of maximum density. Thus, the maximum number derived from the decrement occurs at 360°K, while the maximum number obtained from the modulus data occurs at approximately 320°K. According to the simple Granato-Lücke theory, these curves should be coincident; it is apparent that they are not. It seems probable that the noncoincidence is associated with the existence of more than one dislocation component whose contributions to the total decrement and modulus defect are not equal.

⁵D. O. Thompson and V. K. Paré, *J. Appl. Phys.* 31, 528 (1960).

Fig. 13.5. Fraction of Pinning Points as a Function of Temperature Relative to the Maximum Number Observed.

A logically consistent physical interpretation of the results can be made in terms of vacancy clustering and the onset of void formation around dislocations, as suggested by Coulomb and Friedel. Previous work has indicated that the primary radiation-produced defect responsible for dislocation pinning in this temperature range migrates to the dislocations with an energy of motion of about 1.0 eV in the lattice.⁶ This motion energy is thought to be characteristic of the single vacancy in copper. At the 100°C aging temperature following bombardment, the dislocations become supersaturated with vacancies, if it is assumed that thermal equilibrium is attained before bombardment. When the temperature is lowered, some form of precipitation of the excess vacancy concentration is to be expected. Internal-friction experiments are apparently sensitive to this process through the number of pinning points present, so long as the pinning points remain relatively immobile and the stresses are kept small enough so that dislocation breakaway does not occur. Thus, if two isolated vacancies at one temperature precipitate to form a divacancy at another temperature, the internal-friction experiments would count a change of a factor of 2 in the pinning-point ratio between those temperatures.

In Fig. 13.6 are shown the results of fitting the fractional pinning-point decrement curves of Fig. 13.5 to a theoretical second-order process. The

⁶V. K. Paré and D. O. Thompson, *Acta Met.* 10, 382 (1962).

Fig. 13.6. Fit of Decrement Fraction in Fig. 13.5 to Theoretical Second-Order Curves.

upper curve represents the normalized total number of pinning points as a function of the "universal" parameter $\gamma/n_T e^{-E_B/kT}$, and the two other curves are the component numbers of single and double pinning points. γ is a constant, n_T is the defect concentration per lattice site, E_B is the binding energy of the double pinning point, k is Boltzmann's constant, and T is the absolute temperature. Since n_T is expected to be proportional to the neutron dose, it is evident that either increasing the dose or lowering the temperature, or both, corresponds to moving to the left on the abscissa and to smaller values of the parameters. This procedure evidently favors the formation of doubles at the expense of singles. The points shown on the upper curves are the fractional decrement points of Fig. 13.5 fitted to the theoretical curve. There is an arbitrariness in the fitting procedure due to the fact that the pinning-point distribution is known nowhere as to its absolute composition. However, it can be shown that if a second-order curve is to be fitted, the relative fraction of 55% at 200°K in Fig. 13.5 fixes the lower limit of the absolute ordinate in Fig. 13.6 to 91%, corresponding to a lower abscissa limit of about 8. Having selected the coordinates for the initial point, however, all the remaining points of Fig. 13.5 are fixed in Fig. 13.6 by the value of the binding energy. The best fit of the data to the theoretical curve is obtained with starting coordinates at 360°K for the first bombardment of 97% and 30, and a binding energy of 0.298 eV. Open circles are the first bombardment results, and triangles are the results of the second. It is to be noted that, while any one run is obtained as a function of temperature at constant dose, results obtained after increasing doses must fit on the same curve with the same selection of initial points and binding energy. This is the case because of the dose-proportional n_T in the abscissa parameter. Thus, doubling the neutron dose corresponds to halving the abscissa value for the same temperature. It will be seen that the agreement of theory and experiment is indeed excellent. The modulus data can be fitted to the same kind of curve with a somewhat smaller value for the binding energy. Tests of uniqueness have not been performed, so that it is not known whether the results will fit only a second-order curve. Results from the third bombardment do not fit so well as do the first two, and show a systematic deviation at lower temperatures. Results after

the fourth and fifth doses are not even close, as is evident. Thus, the second bombardment is approaching the limit of the simple, second-order behavior in this work.

It should be possible to evaluate the entropy change associated with the second-order process from the values of the abscissa. With the above binding energy of about 0.3 eV and a reasonable value for the average dislocation loop length of 2.5×10^{-4} cm, so that $n_T \sim 1 \times 10^{-4}$ per lattice site, it follows that the change in entropy is 4.4k. While this is not a particularly meaningful number at present, it is felt that its magnitude is of the right order.

It is thought that the results after larger doses may indicate the formation of larger vacancy clusters in which the divacancy may act as a building block as the excess vacancy concentration is increased by bombardment. Analysis of the isothermal anneals indicated in Fig. 13.3 in terms of pinning-point numbers yields an associated activation energy of about 0.95 eV. The physical meaning of this energy is not readily apparent at present, nor is it known as yet whether this energy depends upon the neutron dose and, consequently, upon the excess vacancy concentration.

An analysis of the strain-amplitude-dependent decrement curves has not been completed in terms of the Granato-Lücke theory for amplitude-dependent damping. It is thought, however, that the behavior of these curves is in at least qualitative agreement with the interpretation given above. After small doses, the strain range before dislocation breakaway occurs is decreased with increasing temperature from 200°K, and the small-amplitude decrement is decreased. The combination may indicate that as the temperature is raised, a greater number of "soft" pinning points become available at the expense of fewer "hard" ones. This result would be consistent with the second-order behavior given above. After the larger bombardments, there is no longer any "inversion" of the amplitude-independent decrement, but neither is there any crossing of the amplitude-dependent curves. The "saturation" character of these later curves may indicate that complete breakaway is achieved. Thus, the higher-dose, low-temperature-induced pinning-point distribution appears to be formed with a larger binding energy per pinning point than does the one for the small dose; it consequently requires a higher range of

temperatures ($\sim 400^\circ\text{K}$) to restore it to the pre-low-temperature distribution.

While these experiments are still rather preliminary, they have served to demonstrate the usefulness of internal-friction techniques in the study of details of dislocation-vacancy interactions in a rather quantitative way. It must be realized that the Granato-Lücke theory does not describe completely adequately the internal friction of most copper crystals studied to date, probably due to the presence of two or more dislocation components. The results given here are deduced on the basis of the simple theory, and as such are subject to refinement. This is the best that can be done at present; on the other hand, the fact that reasonable results are obtained may indicate that the internal friction from one dislocation component is several times larger than that from the others.

Many experiments are indicated as a consequence of the present work. Among them is an extension of the work to higher doses to try to determine whether the annealing, as observed after the fourth and fifth bombardments in the present series, is dependent upon dose. A more complete study of the strain-amplitude curves is also anticipated, as well as completion of the analysis of present ones. These should, in terms of existing theory, enable an evaluation of the relative interaction forces of the various defect clusters with the dislocations. An analysis is difficult, for the validity of existing theory is not certain. Present results indicate, however, that the interaction force of the clustered defect is larger than that of the single, as would be expected.

13.2 ANALYSIS OF INTERNAL FRICTION IN COPPER AT MEGACYCLE FREQUENCIES

V. K. Paré

An experiment was previously reported⁷ in which the internal friction of a copper crystal was measured at 10, 30, 50, 70, 90, and 100 Mc/sec by the ultrasonic pulse method, while the sample was

⁷D. O. Thompson, V. K. Paré, and F. W. Young, Jr., *Solid State Div. Ann. Progr. Rept. Aug. 31, 1960*, ORNL-3017, p 40.

being irradiated at room temperature. The irradiation facility was beam hole HB-2 of the ORR; the fast-neutron flux above 0.75 Mev was about 1.2×10^8 neutrons/cm²/sec.

Although an analysis of the data has been theoretical and experimental results have since become available which make it possible to give a more meaningful treatment. Methods have been developed for applying the results of Oen, Holmes, and Robinson⁸ to the data, and quantitative information has been obtained⁹ on the distribution of delay times required for defects, created by fast-neutron hits, to diffuse to the dislocations and become pinning points. The latter consideration is important, since it implies that the irradiation time is not ordinarily a linear measure of the number of radiation defect pinning points which have arrived at the dislocation lines.

The basic purpose of the experiment is to test the validity of the vibrating-string model of dislocation motion, developed by Koehler¹⁰ and by Granato and Lücke.¹¹ In this model the dislocations are imagined to be held by randomly spaced, fixed pinning points. The free lengths are assumed to oscillate under the influence of the stresses associated with the internal-friction measurement, and the energy loss associated with this motion is calculated by the use of the theory of a vibrating string in a viscous damping medium. The form of the dependence of internal friction on frequency depends on the damping; the situation is analogous to the question of critical damping of a simple harmonic oscillator. The experimental results show clearly that in pure, annealed copper crystals the damping of the dislocation loops (though not of the sample) is greater than "critical." In this case the damping force prevents excitation of the ordinary vibrating-string resonance;¹¹ the logarithmic decrement of the sample is proportional to frequency at low frequencies, where the dislocation motion is limited by line

tension, and inversely proportional at high frequencies, where the damping force limits the motion. These regions are connected by a peak which becomes very broad when one takes into account the assumed random spacing of the pinning points. In treating this case theoretically, one can neglect the inertia of the dislocation line; this has been done by Oen, Holmes, and Robinson.⁸ The effect of irradiation, which introduces additional pinning points, is to simultaneously reduce the magnitude of the peak and move it upward in frequency, in such a way that the asymptotic behavior at high frequency, where the pinning points do not limit the motion, is not affected.

In the study⁹ of delayed pinning by radiation defects, the semiautomatic, elastic constant-internal-friction spectrometer,¹² operating in the neighborhood of 15 kc, was used to study the time dependence of pinning after brief irradiations of a copper crystal at various temperatures from 280 to 370°K. It was found that the results could be interpreted if the existence of both delayed and undelayed pinning points was assumed. The former were found to predominate by a factor of about 150, and their cumulative delay-time distribution, covering a factor of 10^6 in delay time, was derived from the data. The temperature dependence of the delay time was found to be represented by a single activation energy, about 1.0 ev.

With the above information it is possible to calculate, for any irradiation schedule, the actual time dependence of the relative number of pinning points which have arrived at the dislocation lines. The calculation involves integrating over the delay-time distribution; for this purpose the distribution was represented analytically by a piecewise continuous set of power law functions, and the integration was done on the IBM 7090 computer at the Oak Ridge Central Data Processing Facility, with a FORTRAN program written for the purpose. Since it is not yet possible to obtain absolute pinning-point densities, the results are expressed in terms of the equivalent time τ for which, if only the undelayed defects were available, the sample would have to be irradiated to give the pinning-point density actually attained at clock

⁸O. S. Oen, D. K. Holmes, and M. T. Robinson, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1960*, ORNL-3017, p. 3.

⁹D. O. Thompson and V. K. Paré, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p. 24; *Acta Met.* 10, 382 (1962).

¹⁰J. S. Koehler, *Imperfections in Nearly Perfect Crystals*, p. 197, Wiley, New York, 1952.

¹¹A. Granato and K. Lücke, *J. Appl. Phys.* 27, 583, 789 (1956).

¹²D. O. Thompson and F. M. Glass, *Rev. Sci. Instr.* 29, 1034 (1958).

time t . The quantity τ should then be a linear measure of the number of radiation defect pinning points which have arrived at the dislocation lines.

Figure 13.7 shows the frequency dependence of the internal friction at various equivalent times during the irradiation. Clearly, at the start of the experiment the logarithmic decrement is very near to being inversely proportional to frequency; this indicates that the range of measurements is above that of the broad peak. The theoretical function⁸ has been fitted as well as the data permit at each equivalent time; the results are the dashed lines in Fig. 13.7. The passage of the peak through the range of measurement can

Fig. 13.7. Frequency Dependence of Internal Friction in Copper Crystal, at Various Equivalent Irradiation Times. Dashed lines are theoretical curves for a single dislocation component, fitted to the 10- and 70-Mc data.

be seen clearly. Note that the theoretical curves cannot be drawn in with complete arbitrariness; they must all correspond to the same high-frequency asymptotic behavior.

It may be objected that the data appear as a double-humped peak which should be represented by two dislocation components; however, attempts to fit the data in this way have been unsuccessful. On the other hand, the single-component curves shown pass nearly through both the 10- and 70-Mc points. It is therefore felt that the double-hump behavior represents experimental difficulties.

The curves in Fig. 13.7 represent only part of the comparison of theory with experiment. It is also necessary to see whether the pinning-point density implied in the above fit is related in a reasonable way to the input of pinning points to the dislocation lines as indicated by the equivalent time τ . From the theoretical results⁸ it is possible to use the data at a single frequency to obtain values of n_r/n_o , the ratio of the density of radiation defect pinning points to the density of original, preirradiation pinning points; however, in order to do so it is necessary to know f_m , the frequency at which the maximum decrement would have occurred in the preirradiation condition. For consistency a single value of f_m was chosen for the analyses at all six frequencies. Selection was on the basis that the resulting curves of n_r/n_o vs τ should be linear, at least for small τ values. The curves obtained are shown in Fig. 13.8.

The noncoincidence of some of the curves represents the same experimental difficulties which gave rise to the double-hump behavior in the frequency dependence. Note that the 10- and 70-Mc data may be combined to form one continuous curve; this corresponds essentially to the sequence of theoretical curves displayed in Fig. 13.7.

The interesting aspect of the curves in Fig. 13.8 is that one would have expected a straight line of unit slope. Similar curvature has been observed previously¹³ in analogous curves obtained from data taken in the kilocycle range; the interpretation at that time was that there was pinning of two types of dislocations at different rates. The apparent inability to represent

¹³D. O. Thompson and V. K. Paré, *J. Appl. Phys.* 31, 528 (1960).

Fig. 13.8. Variation of Relative Pinning-Point Density with Equivalent Irradiation Time.

the data of Fig. 13.7 by other than one type of dislocation shows, however, that another explanation must be found in the present case. While it is not yet possible to construct a quantitative explanation, it may be noted that the results described in the accompanying article by D. O. Thompson indicate that radiation defect pinning points react with one another while confined to the dislocation lines. It is thus conceivable that some such reaction may tend to remove an increasing fraction of pinning points as the concentration increases, giving rise to the decreasing slope in Fig. 13.8.

The experiment offers the possibility of determining the dislocation parameters in the theory. The average dislocation density in the sample was determined from an etch-pit study by F. W. Young, Jr. From this and the high-frequency asymptotic decrement behavior one can obtain the dislocation damping constant; from a knowledge of f_m and line-tension values derived from the work of DeWit and Koehler,¹⁴ the initial average loop length can be found. Lastly, from the bombarding flux and the initial slope coefficient on Fig. 13.8, one can estimate the number of pinning points per primary knock-on. The results are displayed in Table 13.1. It can be seen that it is somewhat more plausible that the dislocations are of edge type, since the initial

Table 13.1. Parameters Determined for Sample Used in Pulse-Echo Measurements

Dislocation density, cm^{-2}	$L = 1 \times 10^7$	
Damping constant, dyne-sec/cm^2	$B = 1.5 \times 10^{-4}$	
Network length, cm (approximation)	$L^{-1/2} = 3 \times 10^{-4}$	
Frequency of preirradiation maximum, kc	$f_m = 354$	
Assumed dislocation type	Screw	Edge
Line tension, dynes	6.4×10^{-4}	6.4×10^{-5}
Initial loop length, cm	1.2×10^{-3}	3.6×10^{-4}
Total pinning points per primary knock-on	3.6	12

loop length required for screws is greater than a rough estimate of the network length. The value of the damping constant agrees closely with the theoretical estimate of G. Leibfried (quoted by Granato and Lücke¹¹).

13.3 TEMPERATURE DEPENDENCE FROM 250 TO 370°K OF DISLOCATION PINNING IN COPPER SINGLE CRYSTALS BY RADIATION DEFECTS¹⁵

V. K. Paré

D. O. Thompson

Measurements of internal friction and modulus defect at 15 kc have been used to study, in the neighborhood of room temperature, the pinning of dislocations in copper crystals by fast-neutron-induced defects. By measuring at various constant temperatures the delayed pinning following a short irradiation, a delay-time distribution for the pinning defects was obtained, and the process was found to be governed by a single activation energy, 1.0 ev. Although this energy is reasonable for the diffusion of vacancies to dislocations, the shape of the delay-time distribution does not agree with theory, and the apparent number of jumps at the end of the process is anomalously low.

¹⁴G. DeWit and J. S. Koehler, *Phys. Rev.* 116, 1113, 1121 (1959).

¹⁵Abstract of published paper: *Acta Met.* 10, 382 (1962).

13.4 A THERMALLY ACTIVATED INTERNAL-FRICTION SPECTRUM IN COPPER FOLLOWING NEUTRON IRRADIATION¹⁶

D. O. Thompson

Measurements of the internal friction and Young's modulus of a neutron-irradiated copper single crystal as a function of temperature indicate that some conversion of the primary dislocation pinning defect in the range 270 to about 400°K takes place in the dislocation neighborhoods. This conclusion

is based upon the observation that, with the cessation of bombardment and a presently unspecified incubation period, a thermally activated internal-friction spectrum is developed, which is characterized by an energy of about 0.56 ev. These experiments differ from previous work in this field in that an effort is made to allow the dislocation-defect distribution to reach complete thermal equilibrium without the steady-state addition of more defects to the dislocations by continuing bombardment. It is felt that the incubated spectrum may be divacancy-controlled and that the activation energy of 0.56 ev may be interpreted as the motion energy of the divacancy near the dislocations.

¹⁶Abstract of published paper: *Acta Met.* 10, 327 (1962).

14. Ar⁺ Ion Bombardment of Metal Surfaces

A. L. Southern

M. T. Robinson

D. R. Burrowbridge¹

Sputtering yields and atom ejection studies previously reported^{2,3} have been continued with copper, aluminum, and zinc for the better understanding of focusing collisions as postulated by Silsbee⁴ and correlation to the fundamental radiation damage processes. Some of the previous results have been prepared for publication. An abstract of the paper follows.⁵

Sputtering Experiments with 1- to 5-keV Ar⁺ Ions

A. L. Southern

W. R. Willis⁶

M. T. Robinson

Sputtering yields have been determined with 1- to 5-keV Ar⁺ ions normally incident upon targets of type 304 stainless steel, three different types of polycrystalline copper, a wide variety of copper monocrystals, and monocrystals of silicon and germanium. Ejection patterns have been recorded from these targets and from a monocrystal of InSb. The sputtering yield of polycrystalline copper depends on the source of the metal, apparently due to variations in preferred orientation. The yield from copper monocrystals is strongly dependent on orientation, the effect becoming more

pronounced as the energy is increased. A simple model is presented which accounts for this behavior in terms of the variation with direction of the initial mean free path of the incident ion. The ejection patterns leave little doubt that focusing collision chains are primarily responsible for the transport of momentum to the surfaces of close-packed metals.

To determine the number of argon atoms that are interstitial in copper due to argon ions bombarding the copper surface, the following experiment was performed. Samples of polycrystalline 99.999% copper were cut from rolled sheets produced by American Smelting and Refining Company, were etched with nitric acid, and then bombarded with 4-keV argon ions at the same current density. Two samples were bombarded such that 0.396 mg of copper was sputtered from one sample and 1.04 mg from the other. The argon content was determined by vacuum melting and spectrographic analysis of the evolved gas. Only the upper limit of the argon gas could be determined; it was 0.02 μg in the first case and 0.1 μg in the second case. If one considered the argon to be contained only in the volume which is defined by the area of the bombarded spot and the range of argon in copper, then for the first case there would be approximately one argon atom for every ten copper atoms. The different value for the amount of argon contained in the second sample suggests that the argon is not contained within such a volume. Possible explanations would be that the argon diffuses into the undisturbed lattice or that some of the ions lose their energy in a direction with a component parallel to the surface.

Aluminum has been investigated in order to compare the sputtering yield with copper, since the ratio of atomic diameter to lattice spacing

¹Co-op student from Virginia Polytechnic Institute, Blacksburg.

²A. L. Southern *et al.*, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1960*, ORNL-3017, p 52.

³A. L. Southern *et al.*, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 49.

⁴R. H. Silsbee, *J. Appl. Phys.* **28**, 1246 (1957).

⁵Abstract of paper to be published in the *Journal of Applied Physics*.

⁶Professor of Physics, West Virginia Wesleyan College, Buckhannon; consultant.

UNCLASSIFIED
PHOTO 58940 (a) $\langle 100 \rangle$ AT 4 kev (b) $\langle 111 \rangle$ AT 4 kev (c) $\langle 110 \rangle$ AT 4 kevFig. 14.1. Ejection Pattern Obtained from Some Aluminum Monocrystals Under Normally Incident Ar^+ Ion Bombardment.

of aluminum is smaller than that of copper. According to Silsbee,⁴ the efficiency of the focusing process in this case would be less than in copper. Therefore, the sputtering yield would be less, and one would expect the atom ejection spots to be larger. The atom ejection pattern of the (111) plane of aluminum shows the spots to be the base of a cone with a small open angle of $\sim 18^\circ$, whereas in copper this angle was $\sim 12^\circ$. Figure 14.1 shows the ejection patterns from the (100), (111), and (110) planes of aluminum bombarded normal to the surface. The center of the spots corresponds to an ejection angle as expected from the crystal geometry. Preliminary values of the sputtering yield as a function of energy for the (111) plane of 99.99% aluminum, bombarded normal to the surface, are shown in Fig. 14.2. The crystal was electropolished⁷ prior to sputtering. At 5 kev the (111) plane of copper has a yield of 9.3 atoms/ion, and the (111) plane of aluminum has a yield of 2.54 atoms/ion. This value is lower by 60% than that expected from the decrease in focusing efficiency as measured by the spread of the ejected spots. This difference might result from the oxide film on aluminum.

The (0001) plane of zinc was studied to compare the patterns of an hcp crystal that has a staggered chain of atoms with fcc crystals which have close-packed chains of atoms. The atom ejection pattern shows faint spots on the corners of a hexagonal pattern which has an evenly distributed background. This pattern is energy-dependent and has the best focus at a bombarding ion energy of less than 1100 ev. From the crystal lattice spacing and assuming a chain of two atoms only, the ejected atom should escape from the surface at an angle 32° to the normal. With a bombarding ion energy of less than 1100 ev, the outer edges of the pattern were 33° to the normal. Figure 14.3

Fig. 14.2. Sputtering Yield of an Aluminum (111) Plane as a Function of the Normally Incident Ar⁺ Ion Energy.

shows patterns at different bombarding energies for the (0001) plane of zinc. Figure 14.4 shows patterns of (0001) zinc taken at 2 cm from the target for the same bombarding energy at different bombarding times. Spots are detectable in the direction $\langle 2\bar{1}\bar{1}0 \rangle$. The six points appear to be composed of two triangles which can be explained from the zinc model by assuming that each triangular pattern results from atoms ejected from alternate layers.⁸ The background, which can be seen particularly in Fig. 14.4, is due to radiation damage of the crystal.

Experiments are in progress for determining the yields and ejection patterns of ordered and disordered crystals of Cu₃Au; also, the sputtering yields and ejection patterns of gold are being determined.

⁷R. D. Heidenreich, *J. Appl. Phys.* 20, 993 (1949).

⁸C. Lehmann, private communication.

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PHOTO 58941

(a) 1 keV

(b) 1.5 keV

(c) 2 keV

(d) 3 keV

Fig. 14.3. Atom Ejection Pattern for a Zinc (0001) Plane Under Normally Incident Ar^+ Ion Bombardment as a Function of Ion Energy.

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PHOTO 58942

(a) 0.046 amp-sec

(b) 0.08 amp-sec

(c) 0.124 amp-sec

(d) 0.2 amp-sec.

Fig. 14.4. Atom Ejection Pattern for a Zinc (0001) Plane Under Normally Incident Ar^+ Ion Bombardment at 3 kev as a Function of Bombardment Time.

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Part IV. Nonmetals

J. H. Crawford, Jr.

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15. Semiconductor Studies

15.1 RADIATION-INDUCED RECOMBINATION AND TRAPPING CENTERS IN GERMANIUM

I. The Nature of the Recombination Process¹

O. L. Curtis, Jr. J. H. Crawford, Jr.

Extensive measurements on irradiated germanium indicate that previous analyses of the recombination process are incorrect. A model which explains the observations in both *n*- and *p*-type material is presented. According to this model, the recombination level lies 0.36 eV above the valence band in gamma-irradiated *n*-type germanium. Presumably, due to the extensive perturbation caused by neutron irradiation, the level lies slightly lower in neutron-irradiated material. Trapping levels which are not present in antimony-doped germanium occur in arsenic-doped material ~0.17 eV above the valence band. Other trapping levels are observed only in high-resistivity antimony-doped samples. It has not been possible, from the data presented, to determine the values of hole and electron capture cross sections associated with the recombination level; however, the ratio σ_p/σ_n has been determined. Its value is ~1000.

II. Annealing Behavior²

O. L. Curtis, Jr. J. H. Crawford, Jr.

An investigation has been made of the annealing properties of radiation-induced recombination

centers in germanium. The annealing behavior of antimony-doped germanium is grossly different from that of arsenic-doped material. The experimental observations are explained on the following basis: The recombination level at 0.36 eV above the valence band belongs to the vacancy. In antimony-doped material the vacancy disappears by association with an antimony atom. This process does not occur in arsenic-doped material, and higher temperatures are required to produce annealing. It is thought that a trapping level, present only in arsenic-doped material, located 0.17 eV above the valence band might be due to an arsenic-interstitial pair.

15.2 ANNEALING OF IRRADIATION-INDUCED DAMAGE IN GERMANIUM

J. C. Pigg

The annealing properties of the radiation-induced carrier change in antimony-doped germanium have been investigated. The irradiations were conducted at liquid-nitrogen temperatures with Co^{60} gamma irradiation. A model which explains the observed behavior has been proposed. On the basis of this model, the observed annealing consists in vacancy diffusion simultaneously to impurity sites and annihilation centers. An analysis of the activation energy for the annealing process yields values in agreement with vacancy motion but which cannot be resolved into unique components. The complex activation energy is explained by the model in terms of the impurity concentration. It is observed that the change in carrier concentration saturates before complete annealing is achieved. This saturation is stable for further annealing at higher temperatures. The saturation behavior is also explained in terms

¹O. L. Curtis, Jr., and J. H. Crawford, Jr., *Phys. Rev.* **124**, 1731 (1961).

²O. L. Curtis, Jr., and J. H. Crawford, Jr., *Phys. Rev.* **126**, 1342 (1962).

of the model. The vacancies are considered to diffuse to annihilation centers, such as dislocation lines, and to the site adjacent to an antimony atom. Those which go to annihilation centers are removed from the system. Those which go to an antimony atom are trapped. The antimony-vacancy complex can break up to supply vacancies back to the system or can trap an additional vacancy, producing an antimony-vacancy complex. The antimony-divacancy reaction comes into equilibrium very quickly when compared with the annihilation process. As this equilibrium is established, a new energy level associated with the antimony-vacancy complex appears in the forbidden band at 0.09 eV below the conduction band.

A detailed discussion of this work will be issued as a separate report.

15.3 TRAPPING CENTERS IN ARSENIC-DOPED GERMANIUM

O. L. Curtis, Jr.

C. C. Robinson

Recent studies^{3,4} have demonstrated the existence of trapping levels in arsenic-doped germanium which are not observed in material doped with antimony. On the basis of recombination data alone, it is not possible to determine trap concentrations; however, we have been able to obtain this quantity from dc photoconductivity measurements at low temperature. At low temperature the photoconductivity for a given light intensity is increased because the trapping of holes greatly increases the electron lifetime. However, at sufficiently high excitation levels, the traps become filled, and any additional hole-electron pairs quickly recombine. Thus, one expects a break in the photoconductivity vs light intensity curve when the light intensity is just sufficient to fill the traps. Since, as a condition for the experiment, the trapping time is long compared with the recombination time, it can be assumed that all the photoconductivity is contributed by electrons. The number of excess electrons at the light intensity corresponding to

the break in the photoconductivity vs light intensity curve is then equal to the number of hole traps.

Experimentally, a constant current is passed through the specimen and a change in voltage, ΔV , corresponding to the photoconductivity is measured. At low excitation levels, while the traps are being filled, $\Delta V/V \approx \Delta n/n$, where n is the electron concentration in the dark and Δn is the change produced by illumination. (In order to prevent contact effects, only the middle half of the specimen is illuminated. The value $\Delta V/V$ refers only to this central portion.) The value at the break of the curve $(\Delta V/V)'$, is to a good approximation equal to N_t/n . Figure 15.1 illustrates that the expected behavior is observed. Relative intensity is measured by observing the photoconductivity of an unirradiated sample placed beside the test specimen. (The response of the unirradiated sample was determined to be linearly dependent on light intensity in the region of interest by using calibrated light attenuators.) The condition for which a curve such as that in Fig. 15.1 would be expected can be predicted on the basis of a plot similar to that in Fig. 15.3 of the preceding annual progress report (ORNL-3213). From the data of Fig. 15.1, the rate of production of hole traps in arsenic-doped germanium is found to be $\sim 3 \times 10^{-4}$ gamma/cm. This value must be considered tentative since

Fig. 15.1. Photoconductivity as a Function of Intensity for an Arsenic-Doped Germanium Specimen Irradiated with 2×10^{16} gammas/cm². The total voltage drop across the sample was 68 mv.

³O. L. Curtis, Jr., and J. H. Crawford, Jr., *Phys. Rev.* 124, 1731 (1961).

⁴O. L. Curtis, Jr., and J. H. Crawford, Jr., *Phys. Rev.* 126, 1342 (1962).

it is a result of only one measurement. A check will be made to determine whether the number of traps is directly proportional to the total irradiation and whether the trap concentration depends on the amount of arsenic present. Transient measurements will be used to determine capture probability for these centers. Annealing studies are also being performed.

15.4 THERMOELECTRIC POWER IN GERMANIUM

O. E. Schow

An n -type 2-ohm-cm germanium thermoelectric sample and a Hall plate, which were cut from

the same ingot, were measured as a function of photon irradiation at ambient temperature in a Co^{60} gamma field. The experimental conditions were described previously.⁵

A plot of the results of the experiment is shown in Fig. 15.2. The Seebeck coefficient decreased approximately 4% as a result of the first irradiation and then returned to the approximate pre-irradiation value as a result of the two latter exposures. Robinson⁶ made some fast-neutron

⁵O. E. Schow, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, pp 59-60.

⁶C. C. Robinson, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1959, ORNL-2829, pp 129-31.

Fig. 15.2. Change of Sample Parameters due to Co^{60} Photon Irradiation as a Function of Temperature.

irradiations on materials from this same ingot and found a similar behavior of the Seebeck coefficient as a function of irradiation.

Figure 15.3 shows a comparison of the measured Seebeck coefficients of three unirradiated arsenic-doped germanium samples as reported by Geballe and Hull⁷ and the measured Seebeck coefficients of a single antimony-doped germanium sample in which the carrier concentration has been decreased in steps by Co^{60} photon irradiation. The significance of the data is that the Seebeck coefficient should have increased 3.3% for a 40% decrease in the carrier concentration. The measured Seebeck coefficient increased only 0.2%. Since the experimental errors are about 0.2%, it is questionable that the sample changed at all.

In view of the experimental relation between carrier concentration and the magnitude of the Seebeck coefficient, which predicts an increase in the latter, the present observations are difficult to understand.

⁷T. H. Geballe and G. W. Hull, *Phys. Rev.* **94**, 1134 (1954).

Fig. 15.3. Comparison of the Seebeck Coefficient of Irradiated and Unirradiated *n*-Type Germanium as a Function of Carrier Concentration at 280°K.

Since the changes were smaller than expected, the above experiment was scaled up by a factor of 10. The total damage should be approximately 15 times that produced by the 2-ohm-cm irradiation. An analog computer has been constructed to calculate a continuous X-Y plot of the Seebeck coefficient as a function of the sample temperature. Because of the small changes involved in this experiment, a program of rigorous standards has been established.

The materials are now being irradiated.

15.5 RADIATION EFFECTS IN ZINC SULFIDE

R. O. Chester⁸

O. L. Curtis, Jr.

A study of the effects of irradiation of zinc sulfide by reactor neutrons and by Co^{60} gamma rays is being undertaken.

Single crystals have been obtained from the Harshaw Chemical Company. Spectrographic analysis indicates copper (20 ppm) as the major impurity present. Luminescence spectra⁹ verify the presence of copper. Debye-Scherrer diffraction patterns of powder made from the single crystals show only the low-temperature, cubic phase. Laue patterns on single crystals, however, indicate that twinning is present.¹⁰

Bulk resistivity of the crystals is of the order of 10^{10} ohm-cm. Contacts to the sample were produced by applying indium with an ultrasonic soldering iron and heating the sample at 600°C for $\frac{1}{2}$ hr.

It has been suggested¹¹ that the high bulk resistivity of zinc sulfide is due to compensation of donor and acceptor concentrations. Samples with resistivities of the order of 10^2 to 10^3 ohm-cm were prepared by Aven by heating zinc sulfide in the presence of molten zinc. It is argued that zinc acts as a getter for copper and other metallic impurities and removes zinc vacancies.

⁸On leave from the Instrumentation and Controls Division.

⁹We are indebted to C. T. Butler for his kind assistance in obtaining luminescence data.

¹⁰The x-ray studies were performed by M. C. Wittels and F. A. Sherrill.

¹¹M. Aven, *Electronics Div. Abstr.* **11**(1), 46 (May 1962).

Aven verifies the change in copper concentration by observing a well-known¹² change in the luminescence properties of the zinc sulfide.

We have attempted to duplicate Aven's technique. It is felt that zinc sulfide treated in this fashion is likely to be much more useful in radiation-effects studies than the high-resistivity material. Thin layers of the uncompensated material have apparently been produced, and an effort is being made to produce thicker specimens of the lower-resistivity material.

A 24-hr exposure in the Co⁶⁰ source No. 2 (total photon flux of 2.4×10^{16} per square centimeter) produced no observable change in the absorption spectrum of the zinc sulfide crystals. The same was true of a 570-hr exposure in the slant animal tunnel of the ORNL Graphite Reactor (total neutron flux of 3.0×10^{15} per square centimeter). These optical studies are to be followed by more sensitive electrical studies.

15.6 EFFECT OF A PHASE TRANSFORMATION ON THE VAPOR-PHASE GROWTH OF SINGLE-CRYSTAL HgS¹³

O. L. Curtis, Jr.

Attempts have been made to grow single-crystal HgS by a vapor-phase growth technique. The lack of success led to the discovery of a phase transformation which occurs between the temperature of deposition and room temperature. The crystals were deposited in the cubic metacinnabar form and transformed to the hexagonal cinnabar form upon cooling, with a resultant destruction of the deposited crystals. There is a large supercooling effect which seems to depend upon particle size. When cinnabar is heated in an atmosphere of air, the transformation to metacinnabar occurs at $335 \pm 10^\circ\text{C}$. Possibilities for the use of other techniques in the growth of single-crystal HgS are considered.

15.7 GROWTH OF SINGLE-CRYSTAL HgS

O. L. Curtis, Jr.

Efforts are continuing toward the growth of single-crystal cinnabar. It has recently been demonstrated¹⁴ that various crystals can be grown at a temperature below their sublimation

point by adding iodine to the starting material. In our case, the equation for equilibrium is

At the warm ($\sim 420^\circ\text{C}$) end of the tube, the reaction proceeds toward the right. In the cooler ($\sim 300^\circ\text{C}$) portion of the tube, the reverse reaction takes place, releasing the iodine.

Since material is deposited below the phase-transformation temperature,¹⁵ single crystals should be produced. We have succeeded in obtaining small single crystals of HgS. These crystals were bright red and transparent. Unfortunately, the size of the largest crystal ($\sim 2 \times 2$ mm) is inconvenient for electrical measurements. Various alterations in technique are being tried in the hope of producing larger crystals. For instance, the use of chlorine in place of iodine as a transport medium is being planned.

15.8 A STUDY OF THE EFFECTS OF Co⁶⁰ GAMMA RADIATION UPON EXTRINSIC Bi₂Te₃

M. J. Smith

Since most of the properties of Bi₂Te₃ are extremely sensitive to constituent-element ratios,¹⁶ it is difficult to obtain any quantitatively reproducible data from one specimen to the next. Consequently, the band structure and extrinsic conductivity models, which have been proposed for Bi₂Te₃, have been based on qualitative trends. In order to study inherent properties of extrinsic Bi₂Te₃, it is desirable to introduce lattice defects in a specimen, by some controlled method, and to quantitatively evaluate the resultant changes in properties. This philosophy was employed by Schultz *et al.*¹⁷ in their work on heavy deformation and the annealing of Bi₂Te₃. However, most

¹²W. van Gool, *Philips Res. Reports*, suppl 3 (1961).

¹³O. L. Curtis, Jr., *J. Appl. Phys.* **33**, 2461 (1962).

¹⁴R. Nitsche, H. U. Bolsterli, and M. Lichtensteiger, *J. Phys. Chem. Solids* **21**, 199 (1961).

¹⁵O. L. Curtis, Jr., *J. Appl. Phys.* **33**, 2461 (1962).

¹⁶C. B. Satterthwaite and R. W. Ure, Jr., *Phys. Rev.* **108**, 1164 (1957).

¹⁷J. M. Schultz, J. P. McHugh, and W. A. Tiller, Scientific Paper No. 929-8901-P1, Westinghouse Research Laboratories.

of their results were obtained from polycrystalline materials and, because of the extremely anisotropic nature of Bi_2Te_3 , do not necessarily apply as such to single crystalline material.

It has been shown in Groups IV and III-V semiconductors that Co^{60} gamma radiation causes point defects which result in a variation of the Fermi level.¹⁸ The highly reproducible damage of light gamma radiation in these semiconductors does not generally alter lattice parameters, bonding strength, effective mass, or overall band structure. The damage is due primarily to vacancies and interstitials which result in additional doping levels, trapping centers, and variation in current-carrier mobility.

The purpose of this endeavor is to describe and evaluate Co^{60} gamma radiation damage in *p*- and *n*-type Bi_2Te_3 . The results will be used to contribute to the existing knowledge concerning the extrinsic conductivity and band structure of Bi_2Te_3 .

Bismuth telluride crystallizes in a rhombohedral lattice, although, for convenience, it may be indexed as a hexagonal structure.¹⁹ The crystal lattice of the compound is constructed of layers (or leaves²⁰) of atoms lying perpendicular to the major axis of the unit rhombohedral cell. Each layer is composed of atomic sublayers arranged in the sequence $\text{Te}^{\text{I}}\text{-Bi-Te}^{\text{II}}\text{-Bi-Te}^{\text{I}}$. The superscripts are used to distinguish types of differently bonded tellurium atoms.²⁰ The bonding between tellurium atoms in adjacent layers is assumed to be of the Van der Waals²¹ type, indicating an electron density of effectively zero between these bonds. The Bi-Te^{II} bonds are assumed to be fully covalent, whereas there is some degree of ionicity in the Bi-Te^{I} bonds.²⁰ It is assumed that each excess bismuth atom occupies a Te^{II} site and behaves as a singly ionized acceptor, and each excess Tellurium atom substitutes for a bismuth atom and acts as a singly ionized donor.¹⁶ These donor and

acceptor levels lie in the conduction and valence bands, respectively, so that the defects are ionized at all temperatures.²² Airapetiants *et al.*²³ propose that electron conduction proceeds along bismuth layers and that holes move within the tellurium sublattice. Schultz *et al.*¹⁷ report that bismuth and tellurium vacancies (V_{Bi} , V_{Te}) are electrically active defects and are acceptor and donor types, respectively, whereas interstitials are effectively neutral.

Single crystals of Bi_2Te_3 were grown by the Bridgman technique.¹⁶ The use of any foreign doping element was avoided. Current-carrier type and resistivity were controlled by the excess or deficit of tellurium in the melt.¹⁶

Specimens for electrical measurements were cut by an ultrasonic cutter, and leads were soldered in place. Resistivity and Hall measurements were made before and after irradiation by employing the device of Ure.²⁴ The method of Pinnow *et al.*²⁵ was used to obtain thermal diffusivities. All radiations were made at 45°C in the Solid State Division gamma source No. 2.

The observable changes of the electrical properties of *p*- and *n*-type Bi_2Te_3 on a 100-hr radiation in the gamma source are surprisingly large for the high number of current carriers (10^{18} to 10^{19} per cubic centimeter) present in the samples.

The Hall coefficient of *n*-type material at 77°K increases by approximately 20%, after 100 hr irradiation. The percentage of change in the Hall coefficient of *n*-type material vs the initial Hall coefficient (Fig. 15.4) goes through a maximum at $R_0 = 2.5 \text{ cm}^3/\text{coulomb}$. The Hall mobility of *n*-type specimens at 77°K approaches a value of approximately $8.5 \times 10^2 \text{ cm}^2 \text{ v}^{-1} \text{ sec}^{-1}$, after extended gamma radiation. For specimens with initial mobilities above this value, the mobility decreases; for specimens below this value, the mobility increases with time in the source. The resistivity of *n*-type Bi_2Te_3 at 77°K increases by as much as 21% over its preirradiation value after 100 hr gamma radiation. The percentage of change in

¹⁸D. S. Billington and J. H. Crawford, Jr., *Radiation Damage in Solids*, Princeton University Press, Princeton, N.J., 1961.

¹⁹M. H. Franchombe, *British J. Appl. Phys.* **9**, 415 (1958).

²⁰J. R. Drabble and C. H. L. Goodman, *J. Phys. Chem. Solids* **5**, 142 (1958).

²¹J. Black *et al.*, *J. Phys. Chem. Solids* **2**, 240 (1957).

²²B. Yates, *J. Electronics* **6**, 26 (1959).

²³S. V. Airapetiants *et al.*, *J. Tech. Phys. (USSR)*, to be published.

²⁴R. W. Ure, Jr., *Rev. Sci. Instr.* **28**, 836 (1957).

²⁵D. S. Pinnow, Che-yu Li, and C. W. Spencer, *Rev. Sci. Instr.* **32**, 1417 (1961).

Fig. 15.4. Change in Hall Coefficient After 100 hr in Co^{60} Gamma Source vs Initial Hall Coefficient for n -Type Bi_2Te_3 .

resistivity reaches a maximum for specimens of initial mobility of $R/\rho = 1.5 \times 10^3$. There is no noticeable annealing of the radiation damage in n -type material below 150°C .

Bismuth telluride of the p type is exceedingly sensitive to gamma radiation. For p -type specimens with an initial Hall mobility at 77°K of $R/\rho \approx 1.5 \times 10^3 \text{ cm}^2 \text{ v}^{-1} \text{ sec}^{-1}$, the conductivity, Hall coefficient, and Hall mobility go through a sharp minimum, apparently approaching zero, after approximately 100 hr in the source. The crystal remains p type after the minimum. The Hall mobilities of p -type materials approach a value of approximately $7.5 \times 10^3 \text{ cm}^2 \text{ v}^{-1} \text{ sec}^{-1}$ at 77°K upon extended exposures to Co^{60} gamma radiation. There appears to be no appreciable annealing of radiation damage in p -type Bi_2Te_3 at liquid-nitrogen temperatures; however, annealing at room temperature is considerable. Figure 15.5 gives Hall mobility at 77°K vs time of anneal at 300°K for specimen 0-24-121P. Hall mobility is an exponential function of annealing time. The curve reaches a minimum after 5 hr at 300°K and then approaches the unirradiated value in less than 12 hr. All radiation damage appears to be removed upon a 24-hr anneal at 150°C .

Thermal diffusivity increases in n -type and decreases in p -type Bi_2Te_3 upon irradiation. In general, the thermal diffusivity is considerably more sensitive to radiation damage than the electrical properties.

It appears that the magnitude of the radiation effects on p - and n -type Bi_2Te_3 depend, to a large extent, on the percentage of tellurium present. Radiation damage approaches zero for intrinsic material (i.e., tellurium = 60 at. %). This result indicates that the damage involves primarily substitutional atoms (Te_{Bi} in n type and Bi_{Te} in p type). A Frenkel defect^{16,17} involving one substitutional atom reduces the number of

Fig. 15.5. Annealing Curve for Specimen 0-24-121P.

electrons or holes by two in Bi_2Te_3 . Furthermore, an effect upon the electrical properties resulting from a disturbance of the bismuth sublattice in n -type material and the tellurium sublattice in p -type material is consistent with the proposition of Airapetiants *et al.*²³ The interatomic forces in Bi_2Te_3 are much more sensitive to change in the electronegative sublattice than in the electropositive.²⁶ Therefore, the electrical properties are more sensitive to the tellurium vacancy created in p -type material than to the bismuth vacancy in n -type Bi_2Te_3 . It is not understood why radiation damage causes a minimum in the Hall coefficient and electrical conductivity in p -type material. It may be that a significant amount of carrier compensation is created by gamma radiation; however, more experimental verifications are necessary before any conclusion can be drawn.

Cobalt-60 gamma radiation damage in p -type Bi_2Te_3 anneals rapidly at room temperature. The Hall mobility appears to be an exponential function of time. There are two branches to the curve; therefore there are at least two mechanisms involved in the annealing of the damage. One of the mechanisms, perhaps the second branch, represents the diffusion of bismuth interstitials back to tellurium vacancies.

In Bi_2Te_3 crystals with initial Hall mobilities less than $8.5 \times 10^2 \text{ cm}^2 \text{ v}^{-1} \text{ sec}^{-1}$, the Hall mobility increases with increasing doses of gamma radiation. The effect can be attributed to an alteration of the relaxation-time tensor for scattering in the collision term of the kinetic equation.²⁷ The anisotropy of scattering is a function of carrier density,²⁶ and the relation between the Hall coefficient and carrier concentration is a function of the doping level.²⁸ It is seen in Fig. 15.6 that gamma radiation changes the slope of the extrinsic portion of the R vs T^{-1} curve without altering the slope of ρ vs T^{-1} .

²⁶H. J. Goldsmid, *J. Appl. Phys.* **32**, 2198 (1961).

²⁷I. Ya. Korenblit, *Soviet Phys.-Solid State* **2**, 2737 (1961).

²⁸P. A. Walker, *Proc. Phys. Soc. (London)* **76**, 113 (1960).

Fig. 15.6. Hall Coefficient and Resistivity Before and After 300-hr Gamma Radiation vs $1/T$ for Specimen 0-85-88N.

Magneto Resistance of Bi_2Te_3

The fact that an increase in Hall mobility upon extended gamma irradiation of Bi_2Te_3 may be explained by an alteration of the anisotropy of scattering suggested that the galvanomagnetic experiment of Drabble *et al.*²⁹ be repeated for irradiated Bi_2Te_3 . Drabble and Wolfe³⁰ suggested a "six tilted ellipsoids" model for both the conduction and valence bands. The 12 independent galvanomagnetic terms were derived by assuming that a scattering relaxation time τ exists and may be written in the form

$$\tau = bE^{-\lambda}, \quad (1)$$

where the index λ and the quantity b depend on the scattering mechanism.

Korenblit²⁷ repeated the derivation of Drabble and Wolfe but assumed that the collision term in

²⁹J. R. Drabble, R. D. Graws, and R. Wolfe, *Proc. Phys. Soc.* **71**, 3 (1958).

³⁰J. R. Drabble and R. Wolfe, *Proc. Phys. Soc.* **69**, 1101 (1956).

the kinetic equation is obtained for scattering described by a relaxation-time tensor, the components of which are independent of direction of the electron wave vector. The resulting galvanomagnetic coefficients involve an anisotropic scattering term which is not present in the expressions of Drabble and Wolfe.

The effect of irradiation on the magneto-resistance parallel to the cleavage plane is shown in Fig. 15.7 for *n*-type specimen 0-85-88N and *p*-type specimen 0-24-121P. The value of $\Delta\rho/\rho$ increases with time of irradiation for *n*-type material and finally saturates after a change of approximately 10%. For *p*-type material $\Delta\rho/\rho$ initially decreases and then increases continuously upon extended irradiation. If it is assumed that $\Delta\rho/\rho$ is proportional to the mobility squared,³¹ then (where the notation is the same as that of ref 14)

$$\frac{(\rho_{123})^2}{(\rho_{11}\rho_{1133})} = K, \quad (2)$$

³¹I. Eastermann and A. Foner, *Phys. Rev.* **79**, 365 (1950).

where K is a constant. Equation (2) was substantiated by Drabble *et al.*²⁹ for *n*-type material over a rather limited range of doping. However, the results could not be repeated by Goldsmid²⁶ on highly doped *n*-type crystals. Likewise, K appears to be a slowly varying function of gamma radiation dose for *n*-type Bi_2Te_3 . The value of K changes by over one-half an order of magnitude following extended irradiation of *p*-type material. Therefore, preliminary results of the effect of Co^{60} gamma radiation upon the galvanomagnetic properties of Bi_2Te_3 indicate that anisotropic scattering must be considered in the derivation.

15.9 EXPERIMENTAL THRESHOLD IRRADIATION ENERGY REQUIRED FOR THE PRODUCTION OF DEFECT CLUSTERS

J. W. Cleland R. F. Bass

It has long been recognized that the type of lattice defect created by irradiation depends critically on the type and energy of the incident

Fig. 15.7. Magneto-resistance vs Angle Between Current and Magnetic Field.

bombarding particle. The threshold energy for displacement of a lattice atom to a stable interstitial position is about 15 ev for germanium and silicon and 30 to 60 ev for many metals. It has been suggested that the resultant defect is an isolated (point) Frenkel-type interstitial-vacancy pair. One can therefore hope to examine the separate effect of this type of lattice disorder through the use of Co^{60} photon or low-energy (<2-Mev) electron irradiation experiments. The effect of reactor neutrons, fission spectrum neutrons, and monoenergetic 14-Mev neutrons has been shown to be markedly different for certain experimental observations on semiconductors and metals. It has been suggested that the resultant damage is in the form of disordered regions or defect clusters. It would therefore seem important to attempt to ascertain the minimum incident-particle energy or lattice-atom recoil energy required to produce defect clusters; to that end the following experiments have been conducted or are under consideration.

1. The effect of Co^{60} photon irradiation is to introduce defect states that act as acceptors³² in *n*-type germanium, *n* type is converted to *p* type, and it is found that there is an apparent saturation in hole concentration with a limiting Fermi level of 0.24 ev above the valence band after a relatively small total dose (10^{16} photons/cm² for high-purity specimens). Continued irradiation, to doses as high as 10^{19} photons/cm², produces no further change in the hole concentration or Fermi-level position, whereas irradiation with 3×10^{12} fission neutrons/cm² increases the hole concentration and lowers the Fermi level.

2. The critical shear stress of copper single crystals was not increased³³ as a consequence of irradiation with 10^{18} Co^{60} photons/cm², whereas the critical shear stress of copper single crystals has been shown to have a cube-root dependence³⁴ on the total incident flux of 10^{13} – 10^{19} neutrons/cm² from a reactor.

The onset of increase in hole concentration and decrease in the apparent acceptor ionization energy in germanium, and the onset of increase

in critical shear stress in copper single crystals have, therefore, been selected as the criteria to be utilized to determine the threshold energy of a primary recoil for the production of defect clusters in these two types of materials.

3. It has recently been shown³⁵ that lattice defects can be produced in semiconductors from the energetic capture gamma rays that are emitted by cadmium upon thermal-neutron capture. Approximately 25% of these gamma rays have energies in excess of 3 Mev, and these would produce energetic Compton electrons that would impart a primary recoil energy of 300 ev; however, the hole concentration and Fermi-level position of converted germanium is not affected by a total irradiation of 5×10^{15} cadmium gamma rays/cm². The critical shear stress of copper single crystals is also not increased³³ as a consequence of irradiation with capture gamma rays from cadmium.

4. Thermal-neutron-induced (*n*, γ) recoils have been employed^{36,37} to produce lattice defects in materials. One can calculate the maximum kinetic energy of recoil as a consequence of capture gamma-ray emission as 180 ev in germanium, 400 ev in copper, and 780 ev in silicon. Note that these values are greatly in excess of the threshold energy for lattice-atom displacement; however, the end result of (*n*, γ) recoil in germanium and copper is apparently identical with that obtained from Co^{60} photons or cadmium gamma rays.

5. One cannot hope to use conventional radio-isotope photonutron sources in radiation-damage experiments, since the total available flux is too low; however, special sources have been constructed, or could be designed, to provide an intense source of fairly energetic photoneutrons.

(a) Hennelly³⁸ has described an SbBe source with a total output of 10^{10} neutrons/sec from

³²J. W. Cleland, J. H. Crawford, Jr., and D. K. Holmes, *Phys. Rev.* 102, 722 (1956).

³³F. W. Young, unpublished data.

³⁴T. H. Blewitt, to be published in *Nuovo Cimento*; F. W. Young, unpublished data.

³⁵J. W. Cleland, R. F. Bass, and J. H. Crawford, Jr., to be published in the *Journal of Applied Physics*; see also J. W. Cleland, R. F. Bass, and J. H. Crawford, Jr., "Lattice Defect Production in Thermal Neutron Shielded Materials," this report.

³⁶J. H. Crawford, Jr., and J. W. Cleland, "Transmutation Doping and Recoil Effects in Semiconductors Exposed to Thermal Neutrons," vol 1, p 269 in I.A.E.A. Conference on Radioisotopes, Copenhagen, 1960; *Intern. J. Appl. Radiation Isotopes* 9, 189 (1960); J. W. Cleland, to be published in *Nuovo Cimento*.

³⁷R. R. Coltman, to be published in the *Journal of Applied Physics*.

³⁸E. J. Hennelly, *Nucleonics* 19(3), 104, 124 (1961).

1000 curies of antimony, which gives a useful flux of $\sim 10^8$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$. Approximately 70% of the Sb^{124} gamma rays have an energy of 1.74 Mev, and the (γ, n) threshold of beryllium is 1.67 Mev; thus the maximum neutron energy from such a source is 70 kev and the mean energy is 25 to 35 kev. Although the effect of gamma rays is expected to be much greater than that of the much smaller flux of photoneutrons from such a source, the (γ, n) cross section³⁹ in the beryllium is only about 0.8×10^{-27} barn, the qualitatively different effect of the latter should be detectable⁴⁰ if defect clusters were produced. The maximum energy of a recoil in germanium or copper for an incident 25-kev photoneutron is ~ 600 ev. Therefore an SbBe source of the magnitude described by Hennelly³⁸ might be employed to provide a lower-limit check on the minimum recoil energy that is required to produce disordered regions in germanium or increase the critical shear stress of copper. Such an experiment is under way at the present time with the Savannah River SbBe source.

(b) Wattenburg⁴¹ has employed a photoneutron source to measure total cross-section values from 30 kev to about 1.0 Mev for many materials. Metal rods (1.25×5 cm) of manganese, gallium, indium, antimony, lanthanum, or fused NaF were activated in a flux of 10^{12} neutrons $\text{cm}^{-2} \text{sec}^{-1}$ and then placed inside a cylinder of beryllium or D_2O . The actual variation in photoneutron energy in (γ, n) reactions is only 1%; however, this particular experimental arrangement had a total energy variation of about 15%, as measured by proton recoils in a cloud chamber, due to energy losses in the beryllium or D_2O . Representative Q values (neutron emission rate per second) ranged from 2×10^7 to about 1.5×10^6 in these experiments; however, these Q values might be increased by a factor of 200 for the Oak Ridge Research Reactor and by a factor of 3000 to 5000 for the High Flux Isotope Reactor; hence, an irradiation facility of extreme versatility might be designed from these general considerations.

(c) Preliminary experiments have been conducted with an NaF-Be source. Fused NaF was irradiated in an isotope capsule in the No. 1 hydraulic facility of the Oak Ridge Research Reactor in an estimated thermal flux of 2×10^{14} neutrons $\text{cm}^{-2} \text{sec}^{-1}$ for 24 hr, after which the capsule, containing an estimated 280 curies of NaF, was placed inside a $\frac{3}{4}$ in. ID $\times$ $1\frac{3}{4}$ in. OD cylinder of beryllium for 24 to 42 hr of radioactive decay. Ceric sulfate dosimeter⁴² techniques indicated an average value of 1.05×10^5 r/hr, or 1.36×10^{10} photons $\text{cm}^{-2} \text{sec}^{-1}$ for the geometry employed. The atomic displacement cross-section data of Oen and Holmes⁴³ were used to calculate an expected number of lattice-atom displacements from the 2.76- and 1.38-Mev Na^{24} gamma rays (1.04×10^{14} defects/ cm^3 over 24 hr), and the (γ, n) cross-section data³⁹ for beryllium were used to estimate a total 0.82-Mev photoneutron dose of $6 \times 10^{10}/\text{cm}^2$ over 24 hr. High-purity single-crystal n -type samples of germanium were exposed. Figure 15.8 is a plot of log Hall coefficient and resistivity vs inverse temperature before irradiation (curves I) and after successive 24-hr activations and decays (curves II, 42 hr; curves III, 40 hr; curves IV, 24 hr; and curves V, 24 hr). The measured removal of conduction electrons, determined at 78°K, for these exposures at the ambient temperature of the reactor pool were 4.22×10^{13} , 2.66×10^{13} , 2.59×10^{13} , and 2.28×10^{13} per cubic centimeter, respectively. Corresponding data for a similar sample that was exposed to the Na^{24} gammas without the beryllium cylinder gave a measured removal of conduction electrons of 1.99×10^{13} and 2.69×10^{13} per cubic centimeter for two consecutive 24-hr activations and 25-hr decays. One high-purity n -type sample, $n_0 = 6.8 \times 10^{12}$ electrons/ cm^3 at 78°K, was converted to p -type material after the first 42-hr decay and remained a high-resistivity p -type sample, and with an apparent 0.24-ev activation energy, after a total of five successive activations that totaled 170 hr of decay. It is evident from these data that we have constructed a copious gamma-ray source. The removal rate of conduction electrons agrees within about a factor

³⁹E. Guth and M. Mullin, *Phys. Rev.* **76**, 234 (1949).

⁴⁰J. W. Cleland, D. Binder, and J. H. Crawford, Jr., *Solid State Div. Ann. Progr. Rept. Aug. 30, 1956*, ORNL-2188, p 7.

⁴¹A. Wattenberg, *Phys. Rev.* **71**, 497 (1947)

⁴²We are indebted to L. C. Bate of the Analytical Chemistry Division for these measurements.

⁴³O. S. Oen and D. K. Holmes, *J. Appl. Phys.* **30**, 1789 (1959).

Fig. 15.8. Hall Coefficient and Resistivity vs Inverse Temperature for *n*-Type Germanium Before Irradiation and After Successive Exposures in an NaBe Source.

of 4 of that predicted by theory.⁴³ This result is not disturbing in that experimental values are typically low, compared with theoretical values, in most radiation-damage experiments. The gamma-ray effect is also not disturbing, in itself, in that these do not introduce defect clusters. If the calculated dose of photoneutrons is valid, then a total dose of 3.6×10^{11} (0.82-Mev) neutrons/cm² would be expected in these experiments. No evidence of defect clusters was observed, whereas an estimated 3.2×10^{12} neutrons/cm² (fission spectrum) converted *n*-type germanium to *p*-type and indicated the production of defect clusters (see paragraph 9 below).

(d) Molten alkali fluoride salt has been utilized as a heat-exchange medium in certain loop experiments, test reactors, and research or power reactors. The saturation activity of the sodium in

sodium fluoride coolant in the intermediate heat exchanger of the North American Aviation-Atoms International SRE reactor is 0.28 curie/cm³, and the gamma flux has been estimated as 2×10^5 r/hr. It is possible that an extremely potent source of 0.2-Mev (D₂O) or 0.82-Mev (beryllium) photoneutrons could be obtained from such a facility; however, this type of radiation-effects installation has not been critically evaluated for use as a photoneutron source.

(e) Photoneutrons can also be produced from beryllium or D₂O by converting the electrons from a linear accelerator to bremsstrahlung in a high-*Z* converter (i.e., 30 mils of water-cooled gold) and then letting the gamma rays impinge on beryllium or D₂O. Data from Harwell⁴⁴ would indicate a total output of 1.5×10^{19} neutrons/kw-sec for 3.1-Mev electrons on beryllium, 2×10^9 neutrons/kw-sec for 3.1-Mev electrons on D₂O, and 5×10^{10} neutrons/kw-sec for 6.0-Mev electrons on beryllium. Commercial machines are available with an average beam power of 3 to 65 kw; however, the only electron accelerator at ORNL is the small 2.0-Mev Van de Graaff in the Chemistry Division.

(f) It has been suggested that the energetic capture gamma rays from cadmium might be used in a thermal-neutron flux to produce photoneutrons from beryllium or D₂O. An alternative approach is that of Almqvist,⁴⁵ who examined the $\text{Li}^6(n,\alpha)\text{He}^3$ reaction in a thermal-neutron flux, and the subsequent $\text{Li}^6(t,n)\text{Be}^8$ reaction, which produced 2.7×10^{-4} (15.3-Mev) neutron per triton. There is a fundamental limitation concerning photoneutron production in a reactor that should be considered in all radiation-effects experiments. The background fast-(damaging) flux in a thermal-neutron locale can be estimated from the cadmium ratio. For example, the vertical graphite thimbles of the Argonne CP-5 reactor have a thermal flux in excess of 10^{12} neutrons cm⁻² sec⁻¹ and a cadmium ratio in excess of 10^4 ; however, these data would indicate that the background fast (damaging) flux from the reactor itself is about 10^{-3} that of the thermal-neutron flux, whereas the available flux of photoneutrons from beryllium, D₂O, or the $\text{Li}^6(t,n)$ reaction is only about 10^{-4} that of the thermal-neutron flux. The effect of

⁴⁴B. J. Moyer, *Nature* **167**, 346 (1951).

⁴⁵E. Almqvist, *Can. J. Research* **28A**, 433 (1950).

photoneutrons would therefore be totally masked by the fast-neutron background, unless a facility with a cadmium ratio in excess of 10^5 were used. The cadmium ratio of the slant animal tunnel of the ORNL Graphite Reactor is in excess of 10^5 ; however, the thermal flux in this locale is only 1.5×10^9 neutrons $\text{cm}^{-2} \text{sec}^{-1}$.

6. Some preliminary experimental results have been obtained with the $\text{Li}^7(p,n)\text{Be}^7$ reaction, using the 5.5-Mev Van de Graaff⁴⁶ at ORNL. The neutron source is that of a $\frac{1}{4}$ -in.-diam beam of protons that is incident on a rotating target coated with lithium. One must accept an inherent non-uniformity in the neutron flux, since the samples must be located about $\frac{1}{2}$ in. from the target to obtain a reasonable total dose. The lower limit on neutron energy was 0.7 Mev because the machine was unstable for protons with energies lower than 2.42-Mev. The maximum flux of 0.7-Mev neutrons, as determined with a long counter, was about $5 \times 10^7 \text{ cm}^{-2} \text{sec}^{-1}$ for a beam current of $7 \mu\text{a}$. The energy spread was 100 kev. A total irradiation of 10 hr was required to amass an estimated total dose of 1.8×10^{12} neutrons/ cm^2 , which removed 1.6×10^{13} electrons/ cm^3 from a sample of n -type germanium. A total irradiation of 30 to 50 hr under these conditions would be required to convert the purest known n -type germanium to p -type, and this irradiation time would be doubled or tripled for higher-energy neutrons, since the yield decreases with increasing proton energy. One p -type sample of germanium, which had previously been converted from n -type by a total dose of 10^{18} Co^{60} photons/ cm^2 , was also irradiated with an estimated 1.8×10^{12} (0.7-Mev) neutrons/ cm^2 . No evidence of defect clusters or disordered regions was observed.

7. The use of other conventional radioisotope neutron sources for radiation-damage experiments is also limited, at present, by low total fluxes. Such typical commercial sources as Po-Be (90 curies), 2×10^8 neutrons/sec (138-day half-life), or Pu-Be (10 curies), 2×10^7 neutrons/sec (24,000-yr half-life), are not of sufficient intensity. The neutron energy spectrum of this type of source is complex in that the average neutron energy is 4.5 Mev and the maximum neutron energy is

10.8 Mev. One can, however, demonstrate radiation-damage effects⁴⁷ in germanium from an Am^{241} -Be source (16 curies), and one can also predict that Cm^{242} -Be and Cf^{252} -Be sources will be of great value in certain radiation-effects experiments in the future.

8. High-purity n -type samples of germanium were exposed at reactor ambient temperatures (40°C) in the slant animal tunnel of the ORNL Graphite Reactor (1.5×10^9 thermal neutrons $\text{cm}^{-2} \text{sec}^{-1}$) between two $1 \times 1 \times \frac{1}{8}$ in. fission plates that contained 17.16 wt % (2.5 at. %) of 93.16% enriched U^{235} . The estimated flux of fission spectrum neutrons was $3.8 \times 10^{12} \text{ cm}^{-2} \text{hr}^{-1}$. Figure 15.9, which is a plot of log Hall coefficient and

⁴⁷J. W. Cleland and R. F. Bass, unpublished data.

Fig. 15.9. Hall Coefficient and Resistivity vs Inverse Temperature for n -Type Germanium Before Irradiation, After Fission-Spectrum Neutron Irradiation, and After Annealing.

⁴⁶We are indebted to P. H. Stelson and W. T. Newton of the High Voltage Laboratory for assistance in these experiments.

resistivity vs inverse temperature, represents typical data before irradiation (curves I), after three successive 2-hr irradiations (curves II, III, and IV), after a $\frac{1}{2}$ -hr irradiation (curves V), and after a vacuum heat treatment of 20 hr at 450°C (curves VI). The measured removal of conduction electrons as determined at 78°K was 4.18×10^{13} , 3.67×10^{13} , 3.86×10^{13} , and 6.79×10^{12} per cubic centimeter for the four irradiations, corresponding to removal rates of 5.5, 4.8, 5.1, and 3.6 per incident fission spectrum neutron. These values are in reasonable agreement⁴⁸ with -3.2 electrons per neutron as obtained in the Hole 51 fission chamber doughnut of the ORNL Graphite Reactor for an estimated total fast (damaging) flux of 8×10^{11} neutrons $\text{cm}^{-2} \text{sec}^{-1}$. Note that more than 95% of the radiation-induced defects were removed by the vacuum anneal. This value may well be 100%, since the replacement of leads, chemical etch, and remeasurement of sample dimensions may account for the 5% discrepancy. Figure 15.10, which shows plots of log Hall coefficient and resistivity vs inverse temperature, indicates the effect of fission spectrum neutron irradiation on a sample of high-purity *n*-type germanium with an initial electron concentration of $6.6 \times 10^{12}/\text{cm}^3$ (at 78°K) before irradiation (curves I), after three successive 1-hr irradiations (curves II, III, and IV), and after one 2-hr irradiation (curves V). Note that the sample was converted to *p*-type after an estimated total dose of 3.2×10^{12} fission spectrum neutrons/ cm^2 and that the *p*-type hole concentration is increased and the apparent activation energy is decreased by successive irradiations. The measured rate of addition of *p*-type holes was 1.41×10^{12} , 3.91×10^{12} , and 6.6×10^{12} per cubic centimeter, corresponding to an addition rate of 0.37, 1.03, and 0.87 holes per fission spectrum neutron. These values are also in reasonable agreement⁴⁸ with 0.8 hole per neutron as obtained in Hole 51 (see above).

9. High-purity *n*-type samples of germanium have also been exposed to the monoenergetic (14-Mev) neutrons from a $\text{D}(t,n)\text{He}^4$ reaction. These data have been discussed in previous reports⁴⁹

⁴⁸J. H. Crawford, Jr., and J. W. Cleland, *Progress in Semiconductors*, vol 2, p 67, Wiley, New York, 1957.

⁴⁹O. L. Curtis, Jr., and J. W. Cleland, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1959*, ORNL-2829, p 129; *Solid State Div. Ann. Progr. Rept. Aug. 31, 1958*, ORNL-2614, pp 4-6.

Fig. 15.10. Hall Coefficient and Resistivity vs Inverse Temperature for High-Purity *n*-Type Germanium Before Irradiation and After Fission-Spectrum Neutron Irradiation (Now *p*-Type).

and in the open literature,⁵⁰ but not in connection with the current studies of irradiation energy vs nature of damage. Figure 15.11 shows plots of log Hall coefficient and resistivity vs inverse temperature, obtained on a sample identical to that used in Fig. 15.3 for a series of irradiations. Note that 2.6×10^{12} (14-Mev) neutrons/ cm^2 converted the material to *p*-type in a similar manner to that of 3.2×10^{12} (fission spectrum) neutrons/ cm^2 and that the *p*-type hole concentration is increased and the apparent activation energy is decreased by successive irradiations.

In summary, it has been indicated that defect clusters (disordered regions) are apparently introduced in germanium with a total irradiation of

⁵⁰O. L. Curtis, Jr., and J. W. Cleland, *J. Appl. Phys.* 31, 423 (1960).

Fig. 15.11. Hall Coefficient and Resistivity vs Reciprocal Temperature of Initially *n*-Type Germanium After Successive Irradiations of 14-Mev Neutrons.

3×10^{12} fission spectrum or 14-Mev neutrons/cm², whereas no evidence for such defect clusters has been obtained for a total irradiation of 10^{19} Co⁶⁰ photons/cm², 5×10^{15} cadmium gamma rays/cm², 10^{16} (*n*, γ) recoils/cm², 1.8×10^{12} (0.7-Mev) photoneutrons/cm², or 3×10^{11} (0.82-Mev) photoneutrons/cm². Therefore it must be concluded that a neutron energy in excess of 0.7 to 0.8 Mev, which corresponds to a primary recoil energy of 20,000 ev, is required to produce defect clusters or disordered regions, unless a larger total dose is required for this range of neutron energies than has been experimentally available to date.

15.10 LATTICE DEFECT PRODUCTION IN THERMAL NEUTRON SHIELDED MATERIALS⁵¹

J. W. Cleland R. F. Bass J. H. Crawford, Jr.

In connection with our studies of lattice displacements in silicon and germanium caused by

recoil of nuclei upon thermal-neutron capture and capture gamma-ray emission,⁵² it was observed that a comparable amount of lattice damage, as indicated by a change in carrier concentration, was observed even when the specimens were shielded from thermal neutrons by cadmium foil. On the other hand, no detectable change in carrier concentration was observed when a boron-containing shield was used. It is well known that cadmium emits energetic gamma rays upon neutron capture, whereas the dominant capture process of B¹⁰ is an (*n*, α) reaction, with essentially all the α 's being stopped in the shield.

Tabulations of the gamma-ray spectrum resulting from thermal-neutron capture in cadmium have been given by Bartholomew and Higgs⁵³ and Groshev *et al.*,⁵⁴ and these are summarized in Table 15.1. Over one-half the gamma rays emitted fall in the interval 1–5 Mev. Prior experience^{55,56} has shown that Co⁶⁰ gamma rays (1.3 Mev) are sufficiently energetic to displace lattice atoms through the agency of Compton electrons in both silicon and germanium. Moreover, the displacement cross section via this mechanism is expected to increase markedly with gamma-ray energy. This is reflected in the calculations of Oen and Holmes,⁵⁷ whose results for aluminum (*A* = 27) and copper (*A* = 63), obtained for a displacement energy of 15 ev, are shown in Table 15.2. Because of the close similarity of atomic weights of these elements to those of silicon and germanium, the tabulated values should be reasonably representative for

⁵¹J. W. Cleland, R. F. Bass, and J. H. Crawford, Jr., to be published in the *Journal of Applied Physics*.

⁵²J. H. Crawford, Jr., and J. W. Cleland, "Transmutation Doping and Recoil Effects in Semiconductors Exposed to Thermal Neutrons," vol 1, p 269 in *I.A.E.A. Conference on Radioisotopes, Copenhagen, 1960; Intern. J. Appl. Radiation Isotopes* 9, 189 (1960); J. W. Cleland, to be published in *Nuovo Cimento*.

⁵³G. A. Bartholomew and L. A. Miggs, CRGP-784, (1958); see also *Nucleonics* 18(11), 171 (1960).

⁵⁴L. V. Groshev *et al.*, *Atlas of the Gamma Ray Spectra from the Radiative Capture of Thermal Neutrons*, Pergamon, London and New York, 1959.

⁵⁵J. W. Cleland, J. H. Crawford, Jr., and D. K. Holmes, *Phys. Rev.* 102, 722 (1956); J. H. Crawford, Jr., and J. W. Cleland, *J. Appl. Phys.* 30, 1204 (1959).

⁵⁶E. Sonder and L. C. Templeton, *J. Appl. Phys.* 31, 1279 (1960).

⁵⁷O. S. Oen and D. K. Holmes, *J. Appl. Phys.* 30, 1289 (1959).

Table 15.1. Tabulation of Gamma-Ray Spectra from Thermal-Neutron Capture in Cadmium^{53,54}

Gamma-Ray Energy (Mev)	Photons per 100 Captures
0-1	135
1-2	92
2-3	96
3-5	73
5-7	17
7-9	1
9	0.1
Highest 9.05	414.1
Total 410	

Table 15.2. Atomic Displacement Cross Sections in Aluminum and Copper by Gamma Rays⁵⁵

Gamma-Ray Energy (Mev)	$\sigma_d^C \times 10^{-24} \text{ cm}^2$ (Aluminum)	$\sigma_d^C \times 10^{-24} \text{ cm}^2$ (Copper)
1.00	0.33	0.25
1.25	0.50	0.50
1.50	0.68	0.85
1.75	0.85	1.20
2.00	1.00	1.55
2.50	1.30	2.30
3.00	1.50	3.00
4.00	1.90	4.10
5.00	2.10	5.00

the latter when calculated on the same basis. Therefore, simple theory predicts a higher rate of defect introduction for cadmium capture gamma rays as compared with Co⁶⁰ gamma rays.

The observed rates of defect introduction in silicon and germanium by Co⁶⁰ gamma rays, cadmium capture gamma rays, and thermal neutrons are compared in Table 15.3. These values were obtained from the experimental rates of carrier removal ascertained by Hall coefficient measurements in high-purity *n*-type silicon and germanium irradiated at 40°C under the assumption

Table 15.3. Lattice Defect Production in Semiconductors

Type of Irradiation	Material	N_D/ϕ , Theory	N_D/ϕ , Observed
Co ⁶⁰ photons	Silicon	0.026	0.0007
	Germanium	0.023	0.0006
Cadmium gamma rays	Silicon	0.046	0.008
	Germanium	0.076	0.018
Thermal neutrons	Silicon		0.014
	Germanium		0.054

that two carriers are removed per Frenkel pair. All the thermal-neutron and cadmium gamma-ray exposures were conducted in a thermal-neutron facility⁵⁸ of the Oak Ridge Graphite Reactor with a cadmium ratio in excess of 10⁵. The flux of cadmium capture gamma rays was estimated from an expression which represents the flux at the center of a hollow sphere of shield material which is black to thermal neutrons:⁵⁹

$$\phi_\gamma = 3\bar{n}\phi_{th}/8, \quad (1)$$

where $\bar{n}$ is the average number of gamma rays emitted by Cd¹¹³ on thermal-neutron ($\bar{n} = 4, 1$), and ϕ_{th} is the thermal flux to which the enclosing shield is exposed. It is evident that the cadmium gamma rays are ~11 times more effective in producing defects in silicon and ~30 times more effective in germanium for exposures at 40°C than are Co⁶⁰ gamma rays. It is also interesting to note from Eq. (1) that the effect per thermal-neutron capture in the cadmium shield is comparable to the effect per thermal neutron absorbed in the specimen. Also shown in Table 15.3 are the defect introduction rates for the two types of gamma rays as calculated from the displacement cross sections of Oen and Holmes. The cross section employed for the cadmium capture gamma rays is the weighted average obtained from Tables 15.1 and 15.2; all photons with energies greater than 5 Mev, which account for less than 5% of the total spectrum, were neglected. It should be pointed out that the larger

⁵⁸The authors are indebted to W. S. Lyon for his assistance in the determination of thermal flux values.

⁵⁹O. S. Oen, private communication.

theoretical production rates as compared to experimental values are not particularly surprising, since experimental values are typically low by as much as an order of magnitude in radiation-effects experiments. Moreover, at the irradiation temperature employed, appreciable annealing of damage would be expected during the irradiation periods of 100 hr or more employed here.⁶⁰ More significant is the fact that the more energetic capture gamma rays produce relatively much more damage, compared with Co⁶⁰ gamma rays, than the simple theory predicts. This latter observation suggests that either (1) agreement between simple theory and experiment improves, the higher the energy of incident radiation is above the displacement threshold, or (2) the resultant defects from cadmium capture gamma rays are more resistant to thermal anneal than those from Co⁶⁰ gamma rays at the temperature of irradiation. A similar result has been obtained in electron-bombarded germanium,⁶¹ wherein it is observed that the greatest disparity between theory and experiment occurs for electron energies near the displacement threshold.

15.11 EFFECT OF DISLOCATIONS ON Co⁶⁰ RADIATION-INDUCED DEFECTS IN GERMANIUM

J. W. Cleland

R. F. Bass

It has been previously observed⁶² that Co⁶⁰ photons produce randomly distributed interstitials and vacancies that act as acceptors of conduction electrons in *n*-type germanium and that the effect of extended irradiation is to convert *n*-type material to *p* type. In order to investigate the influence of preexisting imperfections, three specimens of widely different dislocation density,⁶³ as determined by etch-pit counting, were irradiated as indicated in Fig. 15.12, which shows a plot

Fig. 15.12. Hall Coefficient and Resistivity vs Inverse Temperature for *n*-Type Germanium Before Irradiation and After Co⁶⁰ Photon Irradiation.

of log Hall coefficient and resistivity vs inverse temperature. The removal rate of conduction electrons was identical, within about 10%, for all three specimens on the *n*-type side, which would suggest that the existing imperfections⁶⁴ had no effect on the irradiation-produced lattice defects. The hole concentration and apparent acceptor level position on the *p*-type side, however, is markedly affected by the initial dislocation density. The Hall coefficient curves saturated for each specimen as indicated, and no further increase in hole concentration or change in the apparent acceptor level was observed as a consequence of continued irradiation; therefore these data would suggest some mode of defect interaction with existing dislocations.

⁶⁰W. L. Brown, W. M. Augustyniak, and T. R. Waite, *J. Appl. Phys.* 30, 1258 (1959).

⁶¹W. L. Brown and W. M. Augustyniak, *J. Appl. Phys.* 30, 1300 (1959).

⁶²J. H. Crawford, Jr., and J. W. Cleland, *J. Appl. Phys.* 30, 1204 (1959).

⁶³We are indebted to A. G. Tweet, General Electric Co., for the dislocation-free material and to R. A. Logan, Bell Telephone Laboratory, for the control and plastically deformed specimens.

⁶⁴J. W. Cleland and J. H. Crawford, Jr., *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1959, ORNL-2829, pp 134-36.

15.12. MONITORING TECHNIQUES FOR CAPTURE GAMMA RAYS

J. W. Cleland

The Biological Thermal-Neutron Treatment Chamber, which is usually referred to as the slant animal tunnel, on top of the ORNL Graphite Reactor, has a thermal flux of 1.5×10^9 neutrons $\text{cm}^{-2} \text{sec}^{-1}$ and a cadmium ratio of 10^5 , which corresponds to a background fast (damaging) flux of only about 10^5 neutrons $\text{cm}^{-2} \text{sec}^{-1}$.

The flux of capture gamma rays inside a hollow shield of material that is black to thermal neutrons has been estimated⁶⁵ as $\frac{3}{8} \bar{n} \phi_{\text{th}}$, where $\bar{n}$ is the average number of gammas emitted per thermal-neutron absorption, and ϕ_{th} is the thermal-neutron flux. One would, therefore, expect $\frac{3}{8}(4.1)(1.5 \times 10^9) = 2.3 \times 10^9$ gammas $\text{cm}^{-2} \text{sec}^{-1}$ from cadmium, with a distribution of gamma-ray energies as indicated.⁶⁵ One would also expect $\frac{3}{8}(1.5 \times 10^9) = 5.6 \times 10^8$ (0.52-Mev)

gammas $\text{cm}^{-2} \text{sec}^{-1}$ from B_4C (boron-impregnated plastic).

Ceric sulfate reduction techniques⁶⁶ were employed to obtain an average value of 400 to 800 r/hr without any shield material, 1860 r/hr inside B_4C , and 9200 r/hr inside cadmium. Ferrous sulfate oxidation techniques⁶⁷ indicated an average value of 600 r/hr without any shield material and 7540 r/hr inside cadmium.

These values correspond to a flux of 3.2×10^8 photons $\text{cm}^{-2} \text{sec}^{-1}$ without shielding (assuming an average energy of 1.0 Mev), 1.0×10^9 for B_4C (assuming a 0.52-Mev gamma ray), and 3.4×10^9 for cadmium (assuming an average energy of 1.25 Mev). It is evident from these data that the rate of production of capture gamma rays is about as predicted and that chemical dosimetry techniques can be employed to determine the photon flux inside a thermal-neutron shield in the absence of fast neutrons.

⁶⁵J. W. Cleland, R. F. Bass, and J. H. Crawford, Jr., "Lattice Defect Production in Thermal-Neutron Shielded Materials," this report.

⁶⁶We are indebted to L. C. Bate of the Nuclear Analysis Group of the Analytical Chemistry Division for these measurements.

⁶⁷We are indebted to C. J. Hochanadel of the Chemistry Division for these measurements.

16. Insulating Crystals

16.1 F- AND M-BAND COLORING IN HEAVILY IRRADIATED HIGH-PURITY KCl

E. Sonder

W. A. Sibley

In the hope of elucidating the processes of radiation coloring in the alkali halides, a comprehensive study of gamma-ray and electron damage in KCl was initiated. The initial goals were (1) to determine the factors that accelerate or slow down the coloring rate, (2) to investigate the relation between F- and M-centers, and (3) to look for any effect of direct knock-on damage by comparing gamma-ray and electron coloring. An abstract of the results of gamma irradiations, which will appear in the *Physical Review*,¹ follows:

Measurements of F- and M-band absorption at room temperature as a function of gamma irradiation are used to study the relation between F-centers and M-centers in a number of KCl crystals from a variety of sources. Although the F-center coloring curves exhibit a variety of forms, presumably due to different trace impurities and imperfections in the crystals, the M-center density seems in all cases to be nearly proportional to the square of the F-center concentration. The ratio of the M-center concentration to the square of the F-center concentration appears to be related to trace impurity content. Zone refining decreases the relative M-center introduction, as does deliberate doping with calcium, whereas plastic deformation has no effect on the ratio. Bleaching increases the M-center concentration at the expense of F-centers; however, short reirradiation with gamma rays causes the ratio of M-center concentration to the square of F-center concentration to return to its pre-bleaching value. The results are thought to indicate that a radiation equilibrium exists between F- and M-centers which is frozen-in when the crystals are removed from the source.

Effect of Radiation Intensity

The fact that the M-center concentration is approximately proportional to the square of the F-center concentration in a given sample of KCl has been demonstrated previously.^{1,2} Figure 16.1 shows clearly that the ratio or slope of the α_M vs $[\alpha_F']^2$ curve, where α_M is the absorption coefficient at the maximum of the M-band and α_F' is the corrected absorption³ at the peak of the F-band, is quite strongly dependent upon the intensity of

¹W. A. Sibley and E. Sonder, to be published in the *Physical Review* on October 15.

²B. J. Faraday, H. Rabin, and W. D. Compton, *Phys. Rev. Letters* 7, 57 (1961).

³F. Okamoto, *Phys. Rev.* 124, 1090 (1961).

Fig. 16.1. Effect of Radiation Intensity on the F- and M-Center Relation. The dashed curve is for a sample gamma-irradiated in a 4.5×10^6 r source. The solid lines are for samples electron-irradiated with 1.5-Mev electrons and the current densities shown.

irradiation. [In the work of Okamoto,³ dichroic absorption in the *F*- and *M*-bands was measured and indicated that one-half to one times the *M* absorption at 8250 Å also appears as additional *M*-center absorption under the *F*-band. Correction for this was made by subtracting 0.8 times the height of the *M*-band from the height of the *F*-band; that is, $\alpha'_F = \alpha_F$ (measured) - 0.8 α_M .] Each solid curve in the figure is for one run, that is, for a series of absorption measurements performed after successive irradiations with a given electron current. The four samples were all from the same ingot of Harshaw KCl. It is quite clear that, as is the case for gamma irradiation, straight lines are obtained; however, the slopes are smaller; that is, the relative *M*-center concentration is less for irradiations with higher currents. To compare these results with those on gamma-irradiated KCl, a curve for a previously reported gamma-irradiated Harshaw KCl sample² is included as a dashed line.

A comparison between gamma and electron irradiations can be made by calculating the energy absorbed per second for the electron- and gamma-irradiated samples. In the electron case it was assumed that all the energy lost by the slowing electrons caused ionization in the KCl. In the gamma case a predominance of Compton absorption of the 1.2-Mev Co⁶⁰ gamma rays was assumed, so that simple conversion from the 4.5×10^6 r calibration of the source was possible. In Table 16.1 are shown the values of energy absorbed per second as well as the slope for the samples shown in Fig. 16.1.

It might be noted that, as was the case in the gamma-irradiated KCl, the α_M vs $[\alpha'_F]^2$ curves do

not pass through the origin. Since, at the irradiation fluxes used in these experiments, effects due to the early stages of coloring are all concentrated very near the origin of our graphs, it is tempting to attribute the excess *M*-center concentration observed to early-stage processes. The implication would be that some of these early-stage processes might take place in regions of anomalously high *F*-center concentrations, that is, near impurity clusters or dislocations where the tendency to form di-*F*-centers would be greater than in the bulk of the KCl.

Equilibrium Between *F*- and *M*-Centers

It has been reported previously that it is possible after bleaching a sample to cause it to return to its pre-bleaching, relative *F*- and *M*-center concentrations with very little additional radiation.^{1,4} Since different intensities of irradiation caused different ratios of $\alpha_M/[\alpha'_F]^2$ to be observed, a number of experiments were performed in which samples were irradiated at constant intensity until they were heavily colored; then, for short irradiation times, the intensity was changed. In Table 16.2 is presented the radiation history of three such runs, one in which the sample was first colored in the gamma source and two where the primary coloring was done by using the electron accelerator with 0.02 and 0.36 $\mu\text{a}/\text{cm}^2$ respectively. The results are illustrated in Fig. 16.2. The curves of Fig. 16.1 are reproduced

⁴L. E. Silverman and L. I. Grossweiner, *Phys. Rev.* **121**, 1072 (1961).

Table 16.1. Rate of Energy Absorption and Slope of α_M vs $[\alpha'_F]^2$ Plots

Sample	Type of Irradiation	Current Density ($\mu\text{a}/\text{cm}^2$)	Energy Absorbed ($\text{Mev cm}^{-3} \text{ sec}^{-1}$)	Slope (cm)
H4	4.5×10^6 r	Gamma source	1×10^{11}	1.0×10^{-3}
H27	1.5-Mev electrons	0.02	5×10^{11}	7.3×10^{-4}
H23	1.5-Mev electrons	0.06	1.5×10^{12}	4.0×10^{-4}
H21	1.5-Mev electrons	0.18	4.5×10^{12}	2.0×10^{-4}
H22	1.5-Mev electrons	0.34	9×10^{12}	1.2×10^{-4}

Table 16.2. Change in *F*- and *M*-Band Absorption upon Short Irradiations

Sample Designation	Type of Irradiation	Electron Current Density ($\mu\text{a}/\text{cm}^2$)	Irradiation Time	Absorbed Energy (Mev/cm^3)	Corrected Absorption at <i>F</i> -Band Maximum (α_F')	Absorption at <i>M</i> -Band Maximum (α_M)
				$\times 10^{14}$		
H24	Electron	0.33	5.3 min	26.6	159.4	3.54
H24	Electron	0.05	10 sec	0.1	151.8	6.66
H24	Electron	0.05	15 sec	0.2	147.8	8.51
H24	Electron	0.33	10 sec	0.9	160.1	3.50
H26	Gamma		20.5 hr	97.0	98.3	10.69
H26	Electron	0.17	15 sec	0.7	115.4	2.30
H26	Electron	0.17	10 sec	0.4	117.8	2.08
H26	Electron	0.02	60 sec	0.4	112.1	5.86
H26	Gamma		10 min	0.8	102.7	11.20
H27	Electron	0.02	90 min	26.2	80.5	5.14
H27	Electron	0.18	15 sec	0.7	90.3	1.89
H27	Electron	0.18	10 sec	0.4	92.7	1.99
H27	Electron	0.02	60 sec	0.3	88.0	4.80
H27	Gamma		10 min	0.8	78.4	7.76

lightly, and the heavy dashed lines and arrows show the changes in *F*- and *M*-center concentration induced by the short irradiations. The irradiation time for each step is also shown. It is clear from the results that short irradiation at changed intensity will cause the ratio $\alpha_M/[\alpha_F']^2$ to shift to a position characteristic of that intensity. Increased radiation-energy flux causes a decrease in *M*-center and an increase in *F*-center concentrations, and a lowering of the flux causes the converse to take place. Moreover, there is a definite tendency after short irradiation at a given flux for the ratio $\alpha_M/[\alpha_F']^2$ to approach the value it would have reached if the sample had been irradiated at the given flux all the time. This would seem to confirm that an equilibrium exists under irradiation conditions and that, except for the slow introduction of additional *F*- and *M*-centers, the equilibrium is reversible. The time to reach equilibrium, as shown by the 10- and

15-sec irradiation of sample H24, seems to be of the same order as the time necessary to go through the first stage of coloration in these samples.

Similar measurements were performed on a number of samples from an ingot of chlorine-treated KCl. Although the relative *M*-center concentration in all these samples was lower, the same variation of $\alpha_M/[\alpha_F']^2$ with intensity and the reversibility with short irradiation at changed intensity were found.

Comparison of Different KCl Samples

In Figs. 16.3 and 16.4 are presented coloring curves and α_M vs $[\alpha_F']^2$ curves for a number of differently prepared samples of KCl, all irradiated at the same electron current density. [Samples H, I, and O were obtained commercially from Harshaw, Isomet, and Optovac respectively.

Fig. 16.2. Radiation Equilibrium Between F- and M-Centers. The points connected by the heavy dashed lines show changes in F- and M-center concentrations upon irradiation with different intensities.

Fig. 16.3. Introduction of F-Centers by 1.5-Mev Electrons ($0.18 \mu\text{a}/\text{cm}^2$) in Differently Prepared Samples of KCl.

Sample P21 was pulled from the melt after chlorine treatment,⁵ and sample W21 was zone-refined in chlorine gas.⁶ Except for I21, the samples were cleaved from the same ingots as samples with

⁵The chlorine-treated KCl was supplied by R. Pohl and J. Ashe of Cornell University.

⁶The zone-refined KCl ingot was supplied by J. G. Castle and R. Warren of Westinghouse.

Fig. 16.4. Effect of Different Sample Preparation in the F- and M-Center Relationship.

similar letters that were gamma-irradiated. (See ref 2 and the abstract at the beginning of this section.)] The curves are meant to be compared with Fig. 1, ref 1, in which information on preparation and source of ingots P and W is also given (see ref 1, Table 1). The shapes of the curves are qualitatively the same for the gamma- and electron-irradiation cases. This evidence, together with the fact noted earlier that when the actual energy absorbed per second is taken into account the rate of coloration under gamma rays is consistent with coloring rate for electrons, would indicate that the knock-on displacement type of damage is negligible, as compared with photolytic processes,⁷ during electron irradiation of KCl.

16.2 GAMMA IRRADIATION OF DOPED KCl

W. A. Sibley

C. T. Butler

The investigation of the effect of gamma irradiation on high-purity crystals was extended to some KCl samples which had been purposely doped with calcium, strontium, cadmium, and magnesium. These crystals were analyzed by flame photometry techniques in order to determine the concentration of divalent impurities present. Table 16.3 shows the results of these analyses. The experimental procedures utilized in the investigation were the same as those previously described for the high-purity-materials work (see Sec 16.1).

⁷J. H. O. Varley, *Nature* **174**, 886 (1954).

Table 16.3. List of Samples

Sample	Impurity	Concentration (ppm)
KCl:Sr	Sr	210
	Ca	30
KCl:Cd	Cd	>300
	Ca	<10
KCl:Mg	Mg	350
	Ca	30
Ca 1	Ca	110
Ca 3	Ca	280

The *F*-band growth curves for the doped specimens are illustrated in Fig. 16.5. The increased first-stage coloring in the calcium- and strontium-doped samples has been observed previously^{8,9} and is attributed to the increased number of positive-ion vacancies which must be present in order to preserve electrical neutrality within the crystal. Since the *F*-center is known to be a negative-ion vacancy with an associated electron, it is difficult to understand how an increase in the number of positive-ion vacancies in the lattice, which suppresses the number of anion vacancies, increases the *F*-center production rate. Recently, Crawford and Nelson¹⁰ have shown that it is possible to transform a cation vacancy into an *F*-center by photolysis, thus resolving the difficulty. It should be noted, however, that the growth curves for the cadmium- and magnesium-doped KCl do not exhibit this increased coloring in the first stage, even though considerably more impurity is present in these crystals. This indicates either that there is a difference in the way calcium and strontium, in the one case, and cadmium and magnesium in the other, enter the crystal as impurities or that the interaction of these impurities with positive-ion vacancies is

⁸H. W. Etzel, *Phys. Rev.* **87**, 906 (1952).

⁹J. H. Crawford, Jr., paper presented at the International Conference on Crystal Lattice Defects, Kyoto, Japan, Sept. 7–12, 1962.

¹⁰J. H. Crawford, Jr., and C. M. Nelson, *Phys. Rev. Letters* **7**, 57 (1961).

Fig. 16.5. *F*-Center Coloring Curves for KCl Crystals Containing Different Impurities.

different (assuming that only divalent impurities are responsible for the observed change in coloring properties).

If the number of free positive-ion vacancies is less for crystals doped with magnesium and cadmium and if this deficiency accounts for the depressed first stage in these samples, then it should be possible to freeze-in enough free vacancies by a quench from high temperatures to increase the first-stage coloring in these crystals. Blocks cleaved from calcium- and magnesium-doped ingots were held at 525°C for 10 min and then quickly dropped into cold acetone in an effort to quench in the high-temperature defect configuration. Specimens for irradiation were cleaved from the centers of these blocks. Figure 16.6 shows the

Fig. 16.6. *F*-Center Growth Curves of Treated KCl Crystals.

result of coloring the quenched specimens. It is clear that there is an increase in the first-stage coloring of the quenched samples; however, thermal deformation could possibly cause the observed increase. A magnesium-doped crystal from the same ingot was deformed 2.9% in a $\langle 100 \rangle$ direction, in a toolmaker's vise. A specimen cleaved from this block was then irradiated. The results of this treatment are also shown in Fig. 16.6 and indicate that the thermal deformation would have to be about 3% in order to explain the experimental results. This magnitude of thermal deformation is probably not possible. Also, cleavage cracks are always observed on the surface of the quenched blocks. These probably reduce the total stress on the center of the block from which the irradiation specimens are cleaved.

16.3 INVESTIGATIONS OF COLOR CENTERS IN ALKALI HALIDES AT ORNL: STRUCTURE SENSITIVITY OF COLOR-CENTER PRODUCTION IN GAMMA-IRRADIATED KCl¹¹

J. H. Crawford, Jr.

Investigations of the structure sensitivity of color center formation in γ -irradiated KCl at ORNL are reviewed. Both Stage I and Stage II coloration ranges are sensitive to the origin of the crystals studied. The presence of divalent cation (M^{++}) markedly enhances Stage I and suppresses Stage II relative to nominally pure crystals. The enhancement in Stage I can be explained by a model involving the conversion of an isolated cation vacancy into an F -center and a $[Cl_3^-]$ ion by a radiolytic reaction. Mechanisms by which M^{++} might suppress Stage II coloration are explored. These are: (a) Suppression of the concentration of electrons and holes produced by the radiation field by acting as electron-hole recombination centers, (b) poisoning dislocations as a source of lattice defects, and (c) alteration of the nature of quenched-in defects. None of these possibilities

is wholly satisfactory to explain experimental observations.

16.4 LUMINESCENCE OF ALKALI HALIDES

C. T. Butler

Luminescence spectra of several alkali halides were recorded during and immediately following gamma irradiation at 5×10^6 r/hr.

Figure 16.7 shows the spectra of Isomet NaCl and KCl as well as Harshaw CsI, LiF, and KCl. The telescope used to focus light from the crystal into the spectrometer cut off just below 400 $m\mu$. Thus, the apparent peaking of the luminescence of the various samples near 400 $m\mu$ may not be real; indeed, Cerenkov radiation should produce a very high output below 400 $m\mu$. The longer wavelength peaks, however, are more certain. Note that the CsI light output is roughly 1000 times that of the others. The curves have been corrected for instrument response, and all except the NaCl curve have been partially corrected for absorption under their F bands.

Figures 16.8–16.10 present the afterglow spectra for these same specimens, as well as for two doped KCl samples. The glows decreased rapidly after removal from the gamma source; therefore, the curves which begin at about 10 sec and run to about 35 sec after removal do not present the true spectrum in each case. The half-life for decay of the 435 band in the KCl's was about 50 sec; it was not measured for the other materials. The records have been corrected for instrument spectral response. The spectrum of LiF is the only one needing any F -band correction. The F -band location is indicated.

The 435- $m\mu$ peak is dominant for Harshaw KCl, whereas a many-line spectrum predominates in the Isomet KCl afterglow. The components of the Isomet ultraviolet band are separated by 0.24 eV and possibly arise from cyanate, which is present in some of Isomet's alkali halides. This band could be reexcited with visible light after fading, but the ease with which it could be activated dropped with time after irradiation. It could not be excited at all beyond about 10 min after gamma irradiation.

¹¹ Abstract of paper to be presented at the International Conference on Crystal Lattice Defects, Kyoto, Japan, Sept. 7–12, 1962.

UNCLASSIFIED
ORNL-LR-DWG 73819

Fig. 16.7. Luminescence of Various Alkali Halides Under Gamma Irradiation of 5×10^6 r/hr.

UNCLASSIFIED
ORNL-LR-DWG 73820

Fig. 16.8. Luminescent Afterglow of Several Alkali Halides After Gamma Irradiation at 5×10^6 r/hr.

UNCLASSIFIED
ORNL-LR-DWG 73821

Fig. 16.9. Luminescent Afterglow of Gamma-Irradiated KCl. (a) Isomet KCl, (b) Isomet at lowered sensitivity, and (c) Harshaw KCl.

Fig. 16.10. Luminescent Afterglows of Gamma-Irradiated, Doped KCl. (a) KCl: Cd, (b) KCl: Mg.

16.5 EFFECT OF ENERGETIC SECONDARY ELECTRONS ON THE SCINTILLATION PROCESS IN ALKALI-HALIDE CRYSTALS¹²

A. Meyer

R. B. Murray

Experimental results have previously shown that the scintillation efficiency of alkali halide crystals to heavy particles is not a function of dE/dx alone but is instead composed of a series of discrete functions, one for each incident particle. This paper presents an analysis of these experimental results with attention to the effect of energetic secondary electrons (delta rays) produced by the primary particle. In this treatment the total light emitted per unit path length of the incident particle is the sum of two contributions: one from the highly ionized primary column, and one from those delta rays which escape the primary column and produce light with a high efficiency.

16.6 STUDIES OF THE SCINTILLATION PROCESS IN CsI(Tl)¹³

R. Gwin¹⁴

R. B. Murray

An experimental investigation of the scintillation process in CsI(Tl) crystals has been made. The scintillation response of CsI(Tl) crystals, having various thallium contents, was measured for

excitation of the crystals by monoenergetic gamma rays, protons, and alpha particles. The investigation was made to provide a test of some of the features of a theoretical model of the scintillation process in thallium-activated alkali iodides proposed by Murray and Meyer.

In order to insure that the results obtained in this program would provide a critical test of the scintillation model, special attention was paid to technical effects which could influence the interpretation of the experimental data. For example, the effect of the pulse-analysis time on the relative scintillation response of CsI(Tl) to various charged particles was investigated. In addition, the emission spectra of the CsI(Tl) crystals were measured for excitation by X-rays, protons, and alpha particles. A knowledge of the emission spectra of the crystals is necessary in order to be sure that only the luminescence characteristic of the thallium activator is considered in the analysis of the experimental data.

The results of the investigation have shown that the scintillation efficiency of CsI(Tl) is a continuous function of dE/dx , as assumed in the scintillation model, within the accuracy of the

¹² Abstract of paper to be published in the *Physical Review*.

¹³ Abstract of ORNL-3354.

¹⁴ Loanee from Neutron Physics Division.

experiments when the effect of delta rays is considered. This leads to the conclusion that the light output of CsI(Tl) crystals is, in general, a nonlinear function of the energy of the particle. The shape of the dL/dE versus dE/dx curve for CsI(Tl) crystals has been shown, in this work, to be nearly independent of the thallium content of the crystal. This is in distinct contrast to the prediction of the scintillation model.

An examination of the emission spectra of CsI(Tl) crystals has shown that light is emitted in a band, centered at about 3300 Å, which is characteristic of the emission of the pure crystal, as well as the thallium luminescence. The relative intensity of this with respect to the thallium-activator luminescence increases as the thallium content decreases. It has been suggested in this work that the emission band characteristic of pure CsI crystals is associated with the radiative decay of an iodine molecule ion of the type I_2^- , which has trapped an electron. On this basis it has been suggested that thallium-activator luminescence results from the successive capture of an electron and a hole at a thallium center. The 3300-Å emission band of CsI overlaps the optical-excitation spectrum of CsI(Tl) for thallium-activator luminescence. Some thallium centers in

CsI(Tl) are therefore excited by optical emission in the 3300 Å band.

16.7 SPECIFIC HEAT OF SINGLE-CRYSTAL $MnCl_2$ IN APPLIED MAGNETIC FIELDS¹⁵

R. B. Murray

The specific heat of a single-crystal sample of $MnCl_2$ has been measured, in the liquid helium temperature region, in applied magnetic fields extending up to 7.26 kilo-oersteds. In all cases the magnetic field was applied parallel to an a -axis of the hexagonal cell. The two specific heat maxima, which have been previously reported at 1.81° and 1.96°K in zero field, are associated with antiferromagnetic transitions. Both transition temperatures are found to be field dependent. The upper transition temperature increases with applied field while the lower transition temperature decreases. These results are correlated with neutron diffraction studies of the magnetic structure transitions.

¹⁵Abstract of a paper submitted to the *Physical Review*.

17. Pure Materials

17.1 INTRODUCTION

J. W. Cleland

It is the purpose of this program to produce research-quality materials that are needed for the AEC materials research program. An extensive program is being conducted to develop techniques to produce high-purity and controlled-impurity research specimens (mostly single crystals) of immediate and long-range interest to investigators at this laboratory, other AEC installations, and AEC contractors. The actual choice of research materials has been made, for the most part, on the basis of the potential gain to a fundamental understanding of solids; this should be realized through an intensive investigation of a given material by a number of research groups under contract to the AEC. Those materials that are commercially available, for the most part, are not being considered in this program.

The Pure Materials Program involves a number of research divisions at ORNL, and subcontracts have been negotiated with research groups outside ORNL to secure specialized skills relating to certain materials of interest. Three distinct phases are usually required for each material: (1) initial purification of the starting material, (2) crystal growth, and (3) evaluation of the end product. Special analytical techniques, such as mass spectral analysis, neutron activation analysis, and microspectroanalysis, supplied by the Analytical Chemistry Division, are important for the initial purification and for ascertaining the purity of the end product. Many of the actual crystal growth techniques are performed by groups in the Reactor Chemistry, Metals and Ceramics, and Solid State Divisions, and the experience of these groups is required for a final analysis of the crystal perfection.

Approximately 50 in. of $\frac{1}{4}$ -in.-diam, high-purity, high-perfection tungsten single crystals have been

obtained from the Linde Company, Indianapolis, Ind., under contract. These crystals were approximately 2 to 12 in. long and were equally divided between (100), (110), and (111) directions within 10 to 60 min of arc with essentially no lineage. Neutron activation analysis results performed at ORNL indicated <0.7 ppm copper, <30 ppm molybdenum, and <5 ppm tantalum. Low-temperature resistivity ratio measurements conducted in the Metals and Ceramics Division also indicated superior crystals. The tungsten contains <50 ppm total impurities, with <5 ppm each of carbon, oxygen, nitrogen, and sulfur.

The effects of initial surface preparation, crystal orientation, oxygen pressure and oxidation temperature have been critically examined as a part of a program to produce good-quality single crystals of Cu_2O by the Virginia Institute for Scientific Research, Richmond, Va. Unidirectional oxidation has been achieved in specimens up to 1.5 mm thick, and test samples have been submitted to ORNL for further examination.

Large (300-g) single crystals of high-purity Li^7F (99.99% Li^7) have been produced by the Stockbarger technique.¹ Small ($\frac{1}{10} \times \frac{1}{10} \times \frac{1}{2}$ mm) strain-free single-crystal samples of LiF with <100 ppm magnesium have been grown from the gaseous phase. The compound $\text{Na}_2\text{B}_4\text{O}_7$ has been employed as a suitable solvent for TiO_2 and ZrO_2 , and small needles of these materials have been produced. Optically perfect crystals of ThO_2 , 1 mm on an edge, have also been grown. Lithium fluoride has been employed as a suitable solvent for UO_2 , ThO_2 , ZrO_2 , TiO_2 , and MgO . Magnesium oxide crystals (1 mm on an edge) have been produced from LiF-NaF and LiF-NaF-MgF_2 eutectic mixtures.

¹C. F. Weaver, B. J. Sturm, and R. E. Thoma, *Reactor Chem. Div. Ann. Progr. Rept. Jan. 31, 1962*, ORNL-3262, p 187.

A pressurized Bridgman-Stockbarger furnace has been constructed by the Metals and Ceramics Division² and will be used in an attempt to grow Cu_2O crystals directly from the melt. Differential thermal analysis studies of part of the $\text{MgO-Li}_2\text{O-WO}_3$ system were performed in search of a suitable phase region for the growth of MgO . The results are negative. Three modifications of the Verneuil process for crystal growth have been designed and assembled and are now being evaluated as an alternative method for the production of high-melting-point crystals, such as MgO .

17.2 OPTICAL ABSORPTION STUDIES ON Cu_2O

C. M. Nelson³

R. F. Bass

J. W. Cleland

Samples of Cu_2O were supplied under contract to the Solid State Division by the Virginia Institute for Scientific Research for a preliminary investigation of the optical absorption properties.

The following procedure was developed to obtain parallel, polished faces: A flat depression was machined in the end of a circular Bakelite plug to a depth greater than that of the samples. Careful machining was required to keep the face of the depression flat and perpendicular to the sides of the plug. The samples were glued around the edges with Duco cement, after which the faces were ground flat on a lapping machine. The mounted samples were then polished in a Syntron apparatus, using a water suspension of $0-0.3 \mu \text{Al}_2\text{O}_3$ particles. Several samples were also polished using $0-0.1 \mu$ mesh; however, this additional step may not have been necessary. After one side was lapped and polished, the mounted sample was immersed in acetone. One or two hours was required to dissolve the cement, after which the sample was remounted and the other side was lapped and polished. One attempt was made to further reduce the thickness of an already polished sample by further polishing. It was found that the $0-0.3 \mu$ particles had apparently broken down after 2 or 3 hr, so that very little additional material was removed. Number 00 emery cloth was employed to decrease the sample

thickness (only a very few passes were required), after which the sample was repolished with $0-0.3 \mu \text{Al}_2\text{O}_3$. Samples 0.24 to 0.38 mm thick were obtained by the above techniques.

Studies of the initial optical absorption indicated that the first samples submitted and labeled as (111) and (110) had very opaque regions in the interior of the material. The (110) sample was particularly opaque, as shown by the high apparent absorption in the infrared region. There are no resolvable absorption bands from 25,500 to about 6400 Å (the apparent absorption edge) at room temperature. Some absorption is apparent, beginning around 12,000 Å and increasing toward 6400 Å, but no definite bands.

Room-temperature optical absorption measurements were also made on two samples, labeled A-2 and A-3, which had been produced by oxidation from one side only. These samples did not exhibit an opaque region, and no absorption peaks were apparent near the absorption edge at room temperature.

One of the polished (111) samples was remounted, lapped, and polished, on one side only, to less than one-half its original thickness, in an effort to remove any central opaque region. The optical absorption of this sample (0.38 mm thick) compared well with the absorption curve for A-3 (0.345 mm thick). These data would suggest that one must remove the central region from samples originally oxidized on both sides before they can be employed in optical absorption studies.

Sample A-3 was mounted in a Dewar, with paper supports to guard against breakage. Measurements were made after the absorption reached an apparent final value near 80°K (2 hr). A large drop in absorption and change in the edge from near 6400 to 6260 Å was observed. There were some inflection points and changes in slope, but no peaks were apparent for this sample.

It was felt that a yet thinner sample was necessary to investigate absorption at lower temperatures; therefore, sample A-3 was reground and polished from 0.345 to 0.24 mm, and the absorption was determined at room temperature and near 80°K. The edge at room temperature was near 6400 Å. There was again a large drop in absorption and in position of the edge between room temperature and 80°K. The most important features of the measurement near 80°K were the appearance of a sharp peak at 5827 Å and the suggestion of another peak at 5787 Å.

²G. W. Clark, *Metals and Ceramics Div. Ann. Progr. Rept. May 31, 1962*, ORNL-3313, p 181.

³Consultant from Emory and Henry College, Emory, Va.

The A-2 sample, which initially had no opaque regions, was irradiated with Co^{60} photons (No. 1 source). Very little effect was noticed with 1.5 hr of irradiation, but with 4.5 hr and particularly after 20 hr irradiation (2×10^{17} photons/cm²), a decided change was observed near the absorption edge. All measurements were made at room temperature with proper precaution to prevent the sample from being exposed to ambient light after irradiation. There was a noticeable decrease in absorption starting near the edge and continuing as far as was possible with the Cary spectrophotometer (6050 Å). An exposure to the tungsten lamp of the Cary model for a period of about 2 hr restored the absorption near the edge and enhanced it below 6350 Å. No changes during any of these treatments were noticed in the region 25,000 to 6600 Å.

17.3 POTASSIUM CHLORIDE SINGLE CRYSTALS

C. T. Butler

J. R. Russell

The ultimate aim of the KCl pure materials program is to produce, on a routine basis, large single crystals having high chemical purity and low defect concentration. In the first phase of this program, high chemical purity is being sought. Sources for pure starting material, furnace materials, and analysis procedures are being investigated separately.

Starting Material and Furnace-Charge Preparation

While handling and preparation techniques are being developed, reagent-grade material is being used. It is known to contain several metallic and nonmetallic impurities, as well as dissolved gases, organic, and other particulate material. To remove the last two impurities, the KCl powder is recrystallized in quartz or Teflon laboratory ware, using doubly distilled water. It is baked overnight in vacuum before being melted under a dry gas for a pulling run.

In the near future the preparation procedures will be improved by including multiple distillation, zone purification, and/or ingot precasting in a dry HCl atmosphere.

Puller and Zone Refiner

A pulling furnace of the so-called Czochralski type has been constructed. It can be evacuated and can be filled with a static atmosphere. The pull rate is continuously variable from 0.01 to over 2 cm/hr. The vacuum and gas-handling system is stainless steel, whereas the header is nickel-plated stainless steel. The envelope and pull rod are quartz. A nickel pull rod is being made. The seed chuck is monel, nickel, or CA-18 graphite, and the crucibles are CA-18 graphite or platinum. It is too early to determine which materials are best suited to each job. The pulling mechanism is driven from a differential ball screw so that gross changes in seed height can be made without disengaging gears or stopping the slower pulling action.

A horizontal zone refiner has also been constructed. The envelope is of quartz, and the end plates and much of the gas-handling apparatus are of monel. The remainder of the apparatus is Teflon. These materials allow use of HCl gas as the process atmosphere. The crucibles are CA-18 graphite. The zoner will be used both to supply pure, crystalline KCl for experimentation and to purify starting material for the puller.

Evaluation of Boules

Crystals ranging to $3 \times 3 \times 10$ cm have been produced. Routine tests on boules include etch-pit counts, flame photometry tests for calcium, strontium, and magnesium, and spectroscopic runs for many other impurities. Optical absorption spectra are measured from 1μ to the fundamental edge on most boules, and infrared runs are made on some boules to determine the presence of complex-ion impurities.

17.4 RESEARCH MATERIALS INFORMATION CENTER

T. F. Connolly

Introduction

Purpose. — The purpose of the Solid State Division's Information Center is to provide information on methods of production, availability of,

need for, and characterization of high-purity solid-state research materials to both producers and users. This operation will involve two related but separable functions: (1) handling "availability" and "materials required" information and (2) handling scientific reports containing background information on methods and results of assay and characterization, uses, and other significant new developments. These functions will be discussed separately.

Scope. — The materials covered will be *all* high-purity solid-state research materials (e.g., metals, alloys, semiconductors, compounds, elements, solid-solution systems, laser and maser materials, and refractories). The only restriction is that they be research materials — as distinct, for example, from structural materials. Radioactive and stable isotopes (covered by the Isotopes Information Center) will not be included, although there will be some unavoidable overlap: pure charge materials for isotope-separation processes will be within the purview of the Research Materials Information Center (RMIC), as will be certain isotopically enriched pure materials. Material will be characterized according to all physical properties of interest (electrical, magnetic, thermophysical, etc.). An example of the difficulty of setting boundaries, of exactly defining the scope of the Center, is presented by the field of radiation effects. Some parts of this field will have to be included since radiation is used to provide structural information not otherwise available. Irradiation is also a method of chemical analysis (neutron activation) and of producing isotopically enriched materials. It might be argued that it would be desirable to accept the loss of some peripheral information in order to set an arbitrary boundary — to say, perhaps, that radiation effects will be included only when they are involved in the characterization or production of an ultrapure material. Such a boundary, however, is not quite so definite as it sounds (an important characteristic of a material might be how it stands up under irradiation, or an unforeseen future use of some material might be conditional upon its radiation response). Under these circumstances the limit of coverage must be defined by the list of materials itself; radiation effects on high-purity research materials become a necessary part of the collection. Again, the boundary line is at structural materials, which are dealt with by, among others, the Defense Materials Information Center of Battelle Memorial Institute.

Evaluation of Material. — Research personnel, active in their fields, will be depended on for reference and evaluation of reports and papers. A list of reviewers, composed of individuals active in the research-materials field, is being prepared, and individuals selected will be requested to evaluate preprints, papers, and data on available materials and to make recommendations concerning their inclusion in an automated information system. In view of the scientific information problem, this evaluation is a logical and necessary extension of the traditional journal-article reviewing system, an extension based on recognition of the fact that no scientific information system can be effective if the scientist disdains a part in it.

Handling "Availability" and "Materials Required" Information

A data sheet is being used for the listing of materials, noted as produced or requested. Copies of the sheet were sent out (the second week in July) with form letters announcing the establishment of the Center to about 400 commercial industries, research laboratories, universities, government agencies, and other organizations. The response so far has been gratifying. There have been expressions of willingness to cooperate, requests for from 10 to 1000 data sheets, and requests for further information on (1) what is expected from participants in the program, (2) what participants can expect from the program, and (3) details of operation of the program.

Data Sheets and Reprints. — It is hoped, first, that participants will fill out the data sheets (for either produced or desired materials) as completely as possible, even if this involves a delay in response. If the sheets cannot be filled out, the information will be welcomed in any other form. References to pertinent reports or papers are urgently needed, whether they deal with methods of production, assay, tests of use, or evaluations obtained from studies of special physical characteristics. Reprints of such papers would be preferred (their analysis and coding by the RMIC will be discussed in the section on background material), but if they are unavailable, the references will suffice.

Exchange of Information. — Those desiring materials with special physical or chemical characteristics should, of course, describe their needs as fully as possible, with particular

emphasis on the specific inadequacies of available materials. This information will be transmitted to those groups in a position to produce the materials as a guide to the developing needs of their potential users. Person-to-person exchanges or loans of materials will be facilitated through the simple device of tabbed "inverted" files — one for requesters and their requirements and one for materials, their characteristics, and their producers. These files are now being prepared. Every material brought to the attention of the Center as being produced (commercially or in the course of research) or needed will be assigned a card. This card will be inserted in a visible file, and the bottom (or visible) portion will be marked with a colored strip bearing the name of the requester for a special form of the material. Any time an improved or changed material (with its source) is added to a card so tabbed, the information will be checked against the tabbed name or names in the file of requests. If the added material fits the specifications noted in the request file, the requester will be notified of the source. *No purchases, loans, or exchanges will be arranged by the Center.*

Data sheets will be organized, indexed, and stored on 100-ft reels of coded 16-mm microfilm. They will be retrievable through a microfilm viewer-printout combination in answer to specific requests or for inclusion in periodic bulletins.

Handling Background Material

Input. — The background or peripheral material will be taken from several sources, the most important of which are expected to be (1) references and reprints provided with the data sheets and (2) references from program participants which concern significant new developments that come to their attention in their own fields (here, again, reviewers of papers from other sources can be of assistance). Literature searches will be continued routinely by the Center, and special searches are being arranged through the cooperation of the AEC Division of Technical Information Extension at Oak Ridge; ORNL prepublication abstracts are being examined. The Center now receives reports in the categories of Metals and Ceramics, Solid State, Physics, and Reactor Chemistry from all AEC installations. All such material will be evaluated — sometimes merely by considering its source, and, where this is not definitive, by

requesting the review described above. When such a review is requested, the reviewer will be asked, if in his opinion the work is not sufficiently novel, to inform the Center of work that supersedes it.

Coding and Filming. — Evaluated reports will be coded according to a key-term (coordinate-index) system and microfilmed by a special film-encoding camera. They will be retrieved by a code-interpreting viewer-printer by the conventional method of appropriate combinations of assigned numerical equivalents of the key terms.

These terms will be controlled by a thesaurus and limited to items of interest to the program. A tentative list of terms, which is growing, is now at about 400, excluding the names and classes of materials. According to the rate of growth of the list so far and the scope of the Center as now envisioned, the list of terms should not exceed 600. The materials list is now at about 350, and no predictions can be made about its eventual size.

Each paper to be filmed will be analyzed for its key terms, and the numerical code equivalent of its set of terms will appear as coding marks on the film. The film storage and retrieval method may be regarded as a photoelectrically interpreted analog of conventional punched-card systems, with the important difference that repeated sorts of the collection are not necessary to establish a "match" between questions and answers. A typical question so answered would be a survey of the latest preparative procedures for a certain material or class of materials. Answers to questions will be in the form of complete reprints, abstracts, or references — reproduced as photo-prints by an attachment to the automated film scanner. Review articles (as opposed to bulletins) are not planned; however, bibliographies produced by the machine should be good source material for such reviews. When bibliographies are used for this purpose, the user will be asked to provide copies of the review articles for reproduction and dissemination (at cost) to interested program participants.

Present Status

The Center is at its second stage, that of collecting and organizing a mass of information from a wide variety of sources. By October of this year, it should be possible to begin the

feedback process of transmitting "availability" and "materials required" information between producers and users on a routine basis, with some scattered items on production methods.

Collection of documents and references that will form the store of background information for the preparation of surveys and bibliographies has already begun, but this material will not be organized, coded, and microfilmed until the spring of 1963.

The following is a list of program participants to date:

Battelle Memorial Institute
Oak Ridge National Laboratory
Brookhaven National Laboratory
Hanford Laboratories—General Electric Company
U.S. Naval Ordnance Test Station, Inyokern, China Lake, Calif.
U.S. Naval Ordnance Laboratory, White Oak, Silver Spring, Md.
Lewis Research Center, NASA
Cornell University:
Laboratory of Atomic and Solid State Physics
Materials Science Center
The Franklin Institute, Philadelphia, Pa.
Virginia Polytechnic Institute
Iowa State University, Ames Laboratory
Massachusetts Institute of Technology, National Magnet Laboratory
University of Chicago, Institute for the Study of Metals
University of North Carolina
University of California, Berkeley, Lawrence Radiation Laboratories
Catholic University of America
Brown University
University of Pennsylvania, Laboratory for Research on the Structure of Matter
Northwestern University, Materials Research Center
University of Wisconsin
University of Nebraska
Stanford Research Institute, Department of Solid State
Harvard University, Division of Engineering and Applied Physics
Stanford University, Department of Materials Science
Purdue University
Mellon Institute
Atomics International
Sandia Corporation
T. J. Watson Research Center, IBM
Raytheon Company
Sylvania Electric Products, Inc.
Bell and Howell Research Center
Bendix Research Laboratories
Virginia Institute for Scientific Research
Commercial producers such as Texas Instruments, Light and Company, Ltd., Eagle-Picher, Semi-Elements, etc.

17.5 PURIFICATION OF Bi_2Se_3 CRYSTALS DURING AND AFTER GROWTH

M. J. Smith

The extrinsic, electrical properties of chemically pure Bi_2Te_3 (ref 4) and Bi_2Se_3 (ref 5) may be attributed to deviations from stoichiometric proportions. The compound Bi_2Te_3 exists as a single phase over a range of composition through the inclusion of substitutional point defects. That is, excess bismuth atoms occupy tellurium sites and are singly ionized acceptors, and excess tellurium atoms occupy bismuth lattice sites and behave as singly ionized donors.⁴ A similar phenomenon⁵ is considered to be true in Bi_2Se_3 . Selenium acts as an acceptor; however, limited solid solubility of selenium in Bi_2Se_3 prevents growth of *p*-type material from a melt containing excess selenium.⁶

The ability of a crystal to exist as one phase over a range of composition implies some thermodynamic consequences which are important in the preparation and purification of these materials. In Bi_2Te_3 and Bi_2Se_3 the crystal vapor has two phases and two components; thus, it has two degrees of freedom. Therefore, in principle, the composition and, hence, the electrical properties of the crystal can be controlled by regulating the temperature and the partial pressure of one component. In practice, however, it is necessary to work at those temperatures at which the crystal-vapor equilibrium is established in a reasonable length of time.

It has been demonstrated previously that controlled selenium vapor pressures can be used to prevent loss of selenium from Bi_2Se_3 to the vapor at high temperatures.⁷ A device similar to that described in ref 7 was used to attempt vapor-crystal doping of Bi_2Se_3 . The device employed the following modifications: the excess selenium was not held within the specimen chamber but was contained in an additional chamber attached to the specimen chamber by a connecting tube. The temperatures of the selenium chamber, connecting

⁴C. B. Satterthwaite and R. W. Ure, Jr., *Phys. Rev.* **108**, 1164 (1957).

⁵K. Hashimoto, *Mem. Fac. Sci., Kyusyu Univ.* **B2**, 141, 187 (1958); *J. Phys. Soc. (Japan)* **16**, 1970 (1961).

⁶M. J. Smith, unpublished research at Cornell University, Ithaca, N.Y.

⁷M. J. Smith, E. S. Kirk, and C. W. Spencer, *J. Appl. Phys.* **31**, 1504 (1960).

tube, and specimen chamber were controlled separately, thereby regulating the partial pressure of selenium in the system. Resistivity and Hall measurements were taken on a number of single crystals of Bi_2Se_3 at various specimen temperatures and selenium vapor pressures as a function of time.

It can be shown that the approach to equilibrium concentrations within a thin slab is expressed by the following relation:⁸

$$\frac{n_{\text{eq}} - \bar{n}}{n_{\text{eq}} - n_i} = \frac{8}{\pi^2} \sum_{v=0}^{\infty} \frac{1}{(2v+1)^2} e^{-[(2v+1)^2 \pi^2 / b^2] D t}, \quad (1)$$

where n_{eq} is the equilibrium concentration in the crystal of thickness b at ambient temperature T_A , $\bar{n}$ is the average concentration in the crystal at some time t , n_i is the initial concentration at $t = 0$, and D is the diffusion coefficient of the process at the temperature of the specimen.

For $t \times D$, sufficiently large, the first term in the series gives a good approximation:

$$\frac{n_{\text{eq}} - \bar{n}}{n_{\text{eq}} - n_i} \approx \frac{8}{\pi^2} e^{-t D \pi^2 / b^2}. \quad (2)$$

If, in the particular case of Bi_2Se_3 , n_0 is the concentration of current carriers and if this concentration of current carriers is, as proposed, controlled by the vapor pressure of selenium over the crystal,

$$\frac{n_{0\text{eq}} - \bar{n}_0}{n_{0\text{eq}} - n_{0i}} = \frac{n_{\text{eq}} - \bar{n}}{n_{\text{eq}} - n_i} = r \approx \frac{8}{\pi^2} e^{-t D \pi^2 / b^2}. \quad (3)$$

For time sufficiently large, r is independent of the vapor pressure over the crystal and depends only on the ambient temperature, T_A , of the crystal and the time of exposure to a particular vapor pressure of selenium. Hence, r may be calculated as a function of time from Eq. (3) by obtaining several measurements of $\bar{n}_0$ at varying times and vapor pressures of selenium. The natural logarithm of r for specimen CBS-2 is plotted in Fig. 17.1 as a function of time for

Fig. 17.1. Plot of $(n_{\text{eq}} - \bar{n}) / (n_{\text{eq}} - n_i) = r$ vs t for Crystal CBS-2 of Bi_2Se_3 at Several Ambient Temperatures.

various ambient temperatures. The slopes of the straight-line portion of each curve give the diffusion coefficient for the process. Diffusion coefficients obtained on all other specimens of Bi_2Se_3 were identical at higher temperatures. Considerable scatter was encountered at lower temperatures, however, because of the inaccuracy of Eq. (3) at these temperatures.

Figure 17.2 shows a plot of $\ln D$ vs $1/T$. The activation energy of the diffusion process is 3×10^4 cal/mole. The dependence of the diffusion coefficient on temperature may be expressed by

$$D = 3.5 e^{-3 \times 10^4 / RT} \text{ cm}^2/\text{sec}. \quad (4)$$

The self-diffusion coefficient of selenium in Bi_2Se_3 is reported⁹ to be expressed by

$$D_{\text{Se} \rightarrow \text{Bi}_2\text{Se}_3} = 8.5 \times 10^{-9} e^{-5.0 \times 10^4 / RT}. \quad (5)$$

The literature does not contain information on the self-diffusion of bismuth in Bi_2Se_3 ; however, values for the bulk diffusion of antimony in Bi_2Se_3 have been obtained.¹⁰ Antimony is similar to bismuth in size and electronic configuration:

$$D_{\text{Sb} \rightarrow \text{Bi}_2\text{Se}_3} = 1.8 \times 10^{-3} e^{-2.95 \times 10^4 / RT}. \quad (6)$$

⁸W. Jost, *Diffusion in Solids, Liquids, Gases*, 3d ed., p 37, Academic Press, New York, 1960.

⁹A. A. Kuliev and G. B. Abdullaev, *Soviet Phys.-Solid State* 1, 545 (1959).

¹⁰B. I. Boltaks, *Zh. Tekhn. Fiz.* 25, 767 (1955).

Fig. 17.2. Selenium Vapor-Crystal Diffusion Coefficient of Bi_2Se_3 Exposed to Various Vapor Pressures of Selenium vs $1000/T$.

The activation energies of Eqs. (6) and (4) are essentially identical. These results indicate that, if the process is an atomic diffusion phenomenon, it is probably controlled by the diffusion of bismuth rather than selenium. The extremely low diffusion coefficient of selenium would prevent any observable effect on electrical properties in a reasonable length of time. On the other hand, the extremely low solid solubility⁶ of bismuth in Bi_2Se_3 renders control of the electrical properties by bismuth vapor pressures impossible.

The maximum reduction of current carriers which is possible under the conditions studied (ambient

temperature $T_A = 400^\circ\text{C}$; temperature of selenium container $T_{\text{Se}} = 400^\circ\text{C}$) is only 33%; that is,

$$\frac{n_i - n_{\text{eq}}}{n_i} = 0.33 \quad (7)$$

at $T_A = 400^\circ\text{C}$ and $T_{\text{Se}} = 400^\circ\text{C}$. Therefore, it appears that a high degree of purification of Bi_2Se_3 is impossible by means of crystal-vapor doping. However, if the vapor pressure of selenium is controlled over liquid Bi_2Se_3 and a crystal is pulled very slowly from the melt, it is reasonable to predict a reduction in current carriers by several orders of magnitude.

Figure 17.3 is a schematic of a modified Teal-Little crystal furnace designed to maintain varying vapor pressures of constituent elements over a liquid while the crystal is growing. The furnace consists of a flanged quartz pipe (1) which is mounted on a water-cooled base (8). The base supports an auxiliary heater (10). The quartz pipe can be evacuated through outlet (9). Inside the quartz pipe is a stainless steel, wire-wound heater (5). The stainless steel heater tube was machined and grooved, coated with AlO_2 , and wound with B&S 24 Nichrome wire. A viewing window was cut in the heater. The heater element (6) was similarly prepared. Inside the two heaters is a quartz jar (2), which is flanged at the top. A stainless steel top (3) fits into the quartz jar. The stainless steel pulling rod and chuck (not shown) passes through the rod support assembly (11). The inside quartz jar is evacuated through this rod and is sealed to the outside when the stainless steel evacuating tube is lowered into the molten compound in the crucible (7). Furnace (4) maintains an excess of one of the constituent elements at controlled temperatures and thereby controls the vapor pressure of the system.

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Fig. 17.3. Controlled Vapor-Pressure Crystal-Pulling Furnace.

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Part V. Radiation Metallurgy

M. S. Wechsler

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18. Fundamental Studies

18.1 THEORY OF THE DEPENDENCE OF RADIATION-ENHANCED DIFFUSION ON DISPLACEMENT PRODUCTION RATE AND IRRADIATION TEMPERATURE

J. H. Barrett¹

Unless experiments are done at very low temperatures, radiation-produced point defects usually anneal out during the irradiation. The most obvious annealing mechanisms are diffusion of the defects to fixed sinks (dislocations, grain boundaries, and surfaces) and recombination of interstitial-vacancy pairs. The equations for this combination of annealing processes are

$$\frac{dv}{dt} = K - \beta_v \nu_v (v - v_0) - \nu_i v i, \quad (1)$$

$$\frac{di}{dt} = K - \beta_i \nu_i i - \nu_i v i, \quad (2)$$

where K is the displacement production rate (interstitial-vacancy pair production rate) per lattice site, v and i are the concentrations of vacancies and interstitials per lattice site, v_0 is the thermal equilibrium concentration of vacancies, $\beta_v = \alpha_v \lambda^2$, $\beta_i = \alpha_i \lambda^2$, λ = jump distance of a defect, the α 's are concentrations of fixed sinks, and the ν 's are jump frequencies.

Dienes and Damask² have treated the steady state solutions of Eqs. (1) and (2); Lomer³ has treated both the steady state and the transient

solutions of these equations. The solutions below are essentially Lomer's, but they are given in a somewhat more tractable form.

As is the case in many metals, it is assumed here that $\nu_i \gg \nu_v$. In this case, the interstitials will quickly achieve a quasi-steady state. Equation (2) may be solved for i to give

$$i = \left(K - \frac{di}{dt} \right) / \nu_i (\beta_i + v). \quad (3)$$

Except for a very brief initial transient period, it is assumed that $di/dt \ll K$. (A sufficient condition for this assumption is $K/\beta_i^2 \nu_i \ll 1$.) Neglecting di/dt in Eq. (3) and substituting the result into Eq. (1) gives

$$\frac{dv}{dt} = K \frac{\beta_i}{\beta_i + v} - \beta_v \nu_v (v - v_0). \quad (4)$$

On the assumption that $v = v_0$ at $t = 0$, the solution to Eq. (4) is

$$v(t) = v_0 + \left\{ \left[\left(\frac{\beta_i + v_0}{2} \right)^2 + \frac{K\beta_i}{\beta_v \nu_v} \right]^{1/2} - \frac{\beta_i + v_0}{2} \right\} \chi(\beta_v \nu_v t), \quad (5)$$

where $\chi(\tau)$ is defined by

$$(1 - \chi)^{1+\rho} \left(1 + \frac{1-\rho}{1+\rho} \chi \right)^{1-\rho} = e^{-2\tau}, \quad (6)$$

$$\rho = \frac{\beta_i + v_0}{2} \left/ \left[\left(\frac{\beta_i + v_0}{2} \right)^2 + \frac{K\beta_i}{\beta_v \nu_v} \right]^{1/2} \right.$$

¹Theory Section, Solid State Division.

²G. J. Dienes and A. C. Damask, *J. Appl. Phys.* **29**, 1713 (1958).

³W. M. Lomer, *Diffusion Coefficients in Copper Under Fast Neutron Irradiation*, AERE-T/R-1540 (December 1954).

It may be noted that $0 \leq \rho \leq 1$. Limiting forms for $\chi(\tau)$ are

$$\chi = \sqrt{1 - e^{-2\tau}} \quad \text{for } \rho \approx 0,$$

$$\chi = 1 - e^{-\tau} \quad \text{for } \rho \approx 1.$$

For other values of ρ , Eq. (6) may be solved by numerical methods to give $\chi(\tau)$. Figure 18.1 shows a plot of $\chi(\tau)$ for the two limiting cases and one other case. For most radiation damage experiments, K will be large enough to make $\rho \approx 0$.

In studies of radiation-enhanced diffusion, a quantity of interest is the time $t(J)$ required for a certain number of jumps per lattice site to occur. The dependence of $t(J)$ on K and on the absolute temperature T may be expressed in the form

$$t(J) \propto K^p e^{E/kT}. \quad (7)$$

Values of p and E are given in Table 18.1. For completeness the table also includes the simpler cases of either the pair-recombination or the

Fig. 18.1. Plot of χ vs τ for Various Values of ρ .

diffusion-to-fixed-sink annealing processes alone. These latter two processes have been treated by Dienes and Damask.²

It should be pointed out that the above analysis assumes that the point defects are randomly distributed upon irradiation and that they remain

Table 18.1. Values of p and E for the Three Annealing Mechanisms

E_{mv} and E_{mi} are the activation energies for motion of a vacancy and interstitial, respectively. The other symbols are defined in the text.

Annealing Process	Time Range	Type of Defect	p	E
Diffusion to fixed sinks alone	$t \ll (\beta_v \nu_v)^{-1}$	v	$-\frac{1}{2}$	$\frac{1}{2}E_{mv}$
	$t \ll (\beta_i \nu_i)^{-1}$	i	$-\frac{1}{2}$	$\frac{1}{2}E_{mi}$
	$t \gg (\beta_v \nu_v)^{-1}$	v	-1	0
	$t \gg (\beta_i \nu_i)^{-1}$	i	-1	0
Pair recombination alone	$t \ll (K\nu_i)^{-1/2}$	v	$-\frac{1}{2}$	$\frac{1}{2}E_{mv}$
		i	$-\frac{1}{2}$	$\frac{1}{2}E_{mi}$
	$t \gg (K\nu_i)^{-1/2}$	v	$-\frac{1}{2}$	$E_{mv} - \frac{1}{2}E_{mi}$
		i	$-\frac{1}{2}$	$\frac{1}{2}E_{mi}$
Pair recombination and diffusion to fixed sinks ($\rho \approx 0$)	$t \ll 4(\beta_v \nu_v)^{-1}$	v	$-\frac{1}{3}$	$\frac{2}{3}E_{mv}$
		i	-1	0
	$t \gg 4(\beta_v \nu_v)^{-1}$	v	$-\frac{1}{2}$	$\frac{1}{2}E_{mv}$
		i	$-\frac{1}{2}$	$-\frac{1}{2}E_{mv}$

so during their annealing. Another possibly important factor, not embraced by the analysis, is that fixed sinks may be created during the irradiation by a process of vacancy aggregation into small pores which collapse forming dislocation loops. Also, no attempt is made to take the effect of solute-vacancy or solute-interstitial pairing into account. These processes may alter the solute jump frequencies in a significant way.

18.2 EFFECT OF RADIATION ON ATOMIC REARRANGEMENTS IN Cu-Al

J. M. Williams C. Bassani⁴
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Introduction

It has been shown⁶⁻⁸ that the bombardment of Cu-Al alloys with neutrons, electrons, or gamma rays results in a decrease in resistivity, provided the temperature is at once high enough for the radiation-produced defects to be mobile and low enough to be below the temperature where equilibrium order can be obtained by thermal diffusion alone. The decrease in resistivity saturates as the equilibrium arrangement is approached. The phenomenon is illustrated schematically in Fig. 18.2. Curve AB represents the ρ vs T curve observed for normal cooling at a given rate, and curve AC represents the ideal equilibrium curve for an infinitely slow cooling rate. Below T_2 for the normal cooling, the atomic arrangement characteristic of point A is frozen in. Upon isothermal irradiation below T_2 (provided the temperature is not too low), the resistivity decreases from a

Fig. 18.2. Schematic Plot of Resistivity vs Temperature.

point on AB to a point on AC. For single crystals of Cu-15 at. % Al cooled at 15°C/hr, T_2 is about 210°C and the decrease upon irradiation varies from zero at 210°C to about 0.18 microhm-cm at 50°C.⁷

The above theoretical analysis (summarized in Table 18.1, Sec 18.1) shows that a study of the temperature and flux dependence of the rate of radiation-enhanced diffusion yields information concerning the mechanism of annihilation of the diffusion-enhancing defect. The temperature dependence for the reaction in Cu-15 at. % Al has been reported previously;⁷ it was found to correspond to an activation energy of 0.5 ev or about one half the activation energy for motion of vacancies in Cu-17 at. % Al observed by Li and Nowick.⁹ This result suggests that $E = \frac{1}{2}E_{mv}$ in Table 18.1. Preliminary experiments dealing with flux dependence were reported earlier.¹⁰ This work has been extended in the past year. In addition, the effect of repeated irradiation and thermal cycling was studied.

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⁶M. S. Wechsler and R. H. Kernohan, *Phys. Chem. Solids* 7, 307 (1958).

⁷R. H. Kernohan and M. S. Wechsler, *Phys. Chem. Solids* 10, 175 (1961).

⁸M. S. Wechsler and R. H. Kernohan, "The Effect of Radiation on Diffusion-Controlled Reactions in Copper-Base Alloys," paper SM-25/25, Symposium on Radiation Damage to Solids and Reactor Materials, May 7-11, 1962, Venice, Italy.

⁹C. Y. Li and A. S. Nowick, *Phys. Rev.* 103, 294 (1956).

¹⁰M. S. Wechsler et al., *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, pp 32-42.

Neutron Flux Measurements and Calculation of Displacement Production Rate

In the flux-dependence experiments, single crystal samples of Cu-15 at. % Al were irradiated at 100°C at various fluxes (various displacement production rates, K) by using different positions in Hole C of the ORNL Graphite Reactor. The resistivity was measured during irradiation and the time, $\tau_{1/2}$, for half-completion of the decrease in resistivity was taken as a measure of the time, $t(J)$ [Eq. (7), Sec 18.1], for a certain number of atom jumps per site.

In order to determine K for each of the experimental positions, various thermal, resonance, and threshold detectors were used to measure the neutron flux and spectrum as a function of distance into Hole C. The effective threshold energies and the corresponding effective cross sections for several threshold detectors are shown in Table 18.2. The threshold monitors used in Hole C were Np^{237} , U^{238} , Ni^{58} , and S^{32} . The U^{238} fission monitors contained only 2 ppm U^{235} .

The detectors, Co^{59} , Mn^{55} , and Na^{23} , were used in determining the thermal and epithermal flux. Each monitor was irradiated both bare and enclosed in a 0.027-in. thickness of cadmium. The thermal flux is evaluated from the difference

of the induced activities, $A_{\text{thermal}} = A_{\text{bare}} - A_{\text{Cd}}$, using

$$\Phi_{\text{thermal}} = \frac{A_{\text{thermal}}}{\sigma_{\text{th}}},$$

where σ_{th} is the thermal cross section. In order to find the epithermal flux from the activities of the cadmium-covered monitors, we follow a method similar to that given by Trice.¹¹ Assuming a $1/E$ spectral distribution in the epithermal region, we write the activation integral for the cadmium-covered samples as

$$A_{\text{Cd}} = \Phi_0 \left(\int_{E_{\text{Cd}}}^{\infty} \frac{\sigma_{1/\nu}}{E} dE + \int_{E_{\text{Cd}}}^{\infty} \frac{\sigma_r}{E} dE \right) \\ = \Phi_0 (I_{1/\nu} + I_r),$$

where Φ_0 is the flux per unit $\ln E$ interval, E_{Cd} is the cadmium cutoff energy, and $\sigma_{1/\nu}$ and σ_r are the $1/\nu$ and resonance components of the cross sections respectively. The cutoff energy was determined for a cadmium thickness of 0.027 in. in

¹¹J. B. Trice, *Nucleonics* 16(7), 81 (1958).

Table 18.2. Detector Elements and Dosimetry Parameters for Several Threshold Reactions

Detector Element	Nuclear Reaction	E_i , Threshold Energy (Mev)	σ_i , Effective Cross Section (barns)	Reference
Np	$\text{Np}^{237}(n,f)\text{Ba}^{140}$	0.6	1.7	a
U	$\text{U}^{238}(n,f)\text{Ba}^{140}$	1.6	0.58	b
Ni	$\text{Ni}^{58}(n,p)\text{Co}^{58}$	2.9	0.39	b
S	$\text{S}^{32}(n,p)\text{P}^{32}$	3.0	0.32	b
Fe	$\text{Fe}^{56}(n,p)\text{Mn}^{56}$	7.5	0.11	b
Al	$\text{Al}^{27}(n,p)\text{Mg}^{27}$	8.2	0.12	b

^aBased on the Np^{237} fission cross section curve given in H. W. Schmitt and R. B. Murray, *Phys. Rev.* 116, 1575 (1959), and the fission spectrum given in L. Cranberg, *Phys. Rev.* 103, 662 (1956).

^bT. O. Passel and R. L. Heath, *Nucl. Sci. Eng.* 10, 308 (1961).

an isotropic flux, and $I_{1/\nu}$ was evaluated for each monitor. Table 18.3 shows I_r values extracted from recent literature, along with the $I_{1/\nu}$ values and thermal cross sections for the reactions that were used. The analysis of the cadmium-covered activity yields Φ_0 .

The samples were analyzed by the Nuclear Analysis Group of the Analytical Chemistry Division.¹² Gamma counting was used for all activities except for the P^{32} activity, which is due to β emission. The gamma-ray spectrometer consisted of a 3×3 in. NaI crystal and a 200-channel analyzer. For fission monitors, the Ba^{140} activity was separated chemically before counting. The techniques involved in analyzing the samples are presented more fully elsewhere.¹³

The monitors were irradiated simultaneously for a period of about 24 hr with the reactor at full power (3400 kw). The samples were positioned in the hole by placing them in thin plastic bottles and setting these bottles into ports in a $\frac{1}{2}$ -in.-ID $\times$ $\frac{5}{8}$ -in.-OD polyethylene tube which was inserted into the hole. The cadmium-covered detectors were separated from the nearest bare detectors by at least $4\frac{1}{2}$ in. in all cases. The standard error is about $\pm 6\%$ for each of the monitors.

¹²We would particularly like to thank W. J. Hampton for his work in preparing and counting the monitors.

¹³P. Dragoumis, J. R. Weir, and G. W. Leddicotte, *Fast-Flux Measurements in the ORR Core*, ORNL-3028 (1961).

Figure 18.3 shows the results of the study. It is seen that the flux-spectrum is fairly well determined for all positions in Hole C from the center of the reactor out to about $2\frac{1}{2}$ ft into the reflector. The spectrum determined for a position 19 ft 10 in. into the hole is compared with the theoretical spectrum of Robinson, Oen, and Holmes¹⁴ (ROH spectrum) in Table 18.4. The theoretical values (Φ_{theor}) were calculated from the ROH spectrum multiplied by a constant, C , so as to give quantities of the same order of magnitude as the measured values. These results indicate that the application of the ROH spectrum to the calculation of the displacement production rate for this position in the Graphite Reactor is reasonably well justified.

The positions where samples were irradiated are shown in Fig. 18.3. Particularly for the two samples located in the reflector region of the reactor (Positions 5 and 6) where the ratio of thermal to fast flux is high, it was necessary to take into account the displacements produced as a result of (n,γ) recoil events.^{15,16} The displacement production rate due to (n,γ) recoil is

$$K(n,\gamma) = \phi_{\text{th}} \sigma_{\text{th}} \frac{\bar{T}_R}{2T_d}, \quad (1)$$

¹⁴M. T. Robinson, O. S. Oen, and D. K. Holmes, *Nucl. Sci. Eng.* 10, 61 (1961).

¹⁵R. M. Walker, *J. Nucl. Mater.* 2, 147 (1960).

¹⁶R. R. Coltman *et al.*, "Reactor Damage in Pure Metals," to be published.

Table 18.3. Thermal Cross Sections and Activation Integrals Above Cadmium Cutoff Energies for Several Reactions in $1/\nu$ Detectors

Reaction	σ_{th} , Thermal Cross Section ^a (barns)	I_r (barns)	References for I_r	$E_{\text{Cd}} = 0.53$ ev, Cd Thickness = 0.027 in.	
				$I_{1/\nu}$ (barns)	$I_T = I_r + I_{1/\nu}$ (barns)
$\text{Co}^{59}(n,\gamma)\text{Co}^{60}$	37.0	57 ± 4	<i>b, c</i>	16.6 ± 0.5	74 ± 4
$\text{Mn}^{55}(n,\gamma)\text{Mn}^{56}$	13.2	7.9 ± 0.5	<i>b, d, e</i>	5.8 ± 0.2	13.7 ± 0.5
$\text{Na}^{23}(n,\gamma)\text{Na}^{24}$	0.54	0.07 ± 0.01	<i>b</i>	0.23 ± 0.01	0.30 ± 0.02

^aD. J. Hughes and R. B. Schwartz, *Neutron Cross-Sections*, BNL-325 (1958).

^bR. Dahlberg, K. Jirlow, and E. Johansson, *J. Nucl. Energy: Pt. A & B* 14, 53 (1961).

^cF. J. Johnston, J. Halperin, and R. W. Stoughton, *J. Nucl. Energy: Pt. A* 11, 95 (1960).

^dR. Bardes *et al.*, *Trans. Am. Nucl. Soc.* 4, 276 (1961).

^eW. H. Walker, C. H. Westcott, and T. K. Alexander, *Can. J. Phys.* 38, 57 (1960).

Fig. 18.3. Neutron Flux vs Distance into Hole C, ORNL Graphite Reactor.

where ϕ_{th} is shown in Fig. 18.3. The average recoil energy is given by

$$\bar{T}_R = \frac{\overline{E_\gamma^2}}{2Mc^2},$$

where E_γ is the energy of the capture gamma ray and $\overline{E_\gamma^2}$ is a weighted average of E_γ^2 over the capture gamma-ray energy spectrum. The calculation of $K(n, \gamma)$ was made for pure copper, using the capture gamma-ray spectrum given by Troubetsky and Goldstein¹⁷ and the values $\sigma_{th} = 3.8$ barns¹⁸ and $T_d = 25$ ev for the thermal neutron absorption cross section and the displacement energy respectively.

As concerns the calculation of $K(\text{fast})$, the contribution to the displacement production rate made by fast neutron collision events, the *relative* values of $K(\text{fast})$ as a function of position in Hole C may be estimated from the integral flux curves in Fig. 18.3. The integral flux

¹⁷E. Troubetsky and H. Goldstein, *Nucleonics* 18(11), 171 (1960); see also *A Compilation of Information on Gamma-Ray Spectra Resulting from Thermal-Neutron Capture*, ORNL-2904 (May 17, 1960).

¹⁸D. J. Hughes and R. B. Schwartz, *Neutron Cross Sections*, BNL-325 (July 1, 1958).

Table 18.4. Comparison of Calculated and Observed Fluxes for a Position 19 ft 10 in. into Hole C, Graphite Reactor

No.	Monitor	Measured Quantity	Φ_{meas}^a (neutrons cm ⁻² sec ⁻¹)	Φ_{theor}^b (neutrons cm ⁻² sec ⁻¹)	$\frac{\Phi_{\text{meas}}}{\Phi_{\text{theor}}}$
			$\times 10^{10}$	$\times 10^{10}$	
1	Mn ⁵⁵	Φ_0	5.5	4.2	1.3
2	Na ²³	Φ_0	4.4	4.2	1.0
3	Np ²³⁷	$\Phi(E > 0.6 \text{ Mev})$	9.5	9.1	1.0
4	U ²³⁸	$\Phi(E > 1.6 \text{ Mev})$	2.6	3.9	0.67
5	Ni ⁵⁸	$\Phi(E > 2.9 \text{ Mev})$	1.1	1.4	0.78
6	S ³²	$\Phi(E > 3.0 \text{ Mev})$	0.97	1.3	0.75

^aRead from graph in Fig. 18.3.

^bFor Nos. 1 and 2, $\Phi_{\text{theor}} = \Phi_0 = C \left[\lim_{E \rightarrow 0} E \phi(E) \right]$;

for Nos. 3 through 6, $\Phi_{\text{theor}} = C \int_{E_i}^{\infty} \phi(E) dE$, where $\phi(E)$ = ROH spectrum.

above 0.6 Mev was used as determined by the Np^{237} fission threshold detector. In order to set the absolute magnitudes of the values of $K(\text{fast})$, we have relied on the results of Colman *et al.*,¹⁶ who observed that $K(\text{fast}) = 4 K(n,\gamma)$ for copper irradiated at 4°K near the center of the reactor. Using this relation and Eq. (1) for our experiment at the center of the reactor, we may calculate $K(\text{fast})$ for this position and, thus, for all positions in Hole C. Finally, K is set equal to $K = K(\text{fast}) + K(n,\gamma)$, which assumes that no distinction is to be made between the effects of displacements produced by neutron collisions and those resulting from (n,γ) events.

Resistivity Measurements and Discussion

The curves of resistivity vs time of irradiation are given in Fig. 18.4. The change in resistivity is shown normalized to the total decrease, which was approximately the same for each run. The time for half-completion, $\tau_{1/2}$, corresponds to $f = 0.5$. The curves in Fig. 18.4 refer to freshly prepared samples which were annealed at 750°C followed by cooling to room temperature at 15°C/hr. We shall designate these runs as Series

I. Referring to Fig. 18.2, we see that each of the runs in Series I corresponds to a decrease in resistivity from B to C where $T_0 = 100^\circ\text{C}$.

For the runs of Series II, the samples were warmed, following the decrease from B to C (Fig. 18.2), along CD with the reactor on and held at T_2 (210°C) where the original value at A was achieved. Then, with the reactor off, the samples were cooled along AB to $T_0 = 100^\circ\text{C}$. The initial resistivity at B was recovered to within 0.01 microhm-cm on the average, which is about 5% of the total decrease or 0.1% of the absolute resistivity. We may represent the irradiation and thermal cycling treatment as follows (letters refer to points in Fig. 18.2):

$$\begin{array}{ccccccc} & \text{reactor} & & \text{reactor} & & \text{reactor} & \\ & \text{on} & & \text{on} & & \text{off} & \\ B & \xrightarrow{\quad} & C & \xrightarrow{\quad} & A & \xrightarrow{\quad} & B \\ (100^\circ\text{C}) & & (100^\circ\text{C}) & & (210^\circ\text{C}) & & (100^\circ\text{C}) \end{array} \quad (2)$$

The results for repeated runs at position 4 are shown in Fig. 18.5. The first run (Run 4-I) is for a freshly prepared sample. Following Run 4-I, the sample was given the irradiation and thermal cycling treatment of Eq. (2) and Run 4-II was conducted. Then, similar treatments were used for Runs 4-III and 4-IV. For each of the

Fig. 18.4. Fractional Departure from Completion of the Decrease in Resistivity of Cu-Al (15 at. % Al) vs Time of Irradiation at 100°C for Samples at Various Distances into Hole C, ORNL Graphite Reactor.

Fig. 18.5. Fractional Departure from Completion of the Decrease in Resistivity of Cu-Al (15 at. % Al) vs Time of Irradiation for Repeated Runs at Position 4, Hole C, ORNL Graphite Reactor.

Fig. 18.6. Time for Half-Completion of the Decrease in Resistivity as a Function of the Defect Production Rate in Cu-Al (15 at. % Al). The dashed line was obtained from previous electron and gamma irradiation experiments (refs 8, 10).

runs, the magnitude of the decrease in resistivity was about the same. However, as Fig. 18.5 shows, the effect of the cycling treatment is to increase $\tau_{1/2}$ for Run 4-II by a factor of 2.5 over that for Run 4-I. The repeated cycling treatment for Runs 4-III and 4-IV did not appreciably increase $\tau_{1/2}$ further.

In Fig. 18.6 the values of $\tau_{1/2}$ are plotted vs K for Series I and II. For both series, $\tau_{1/2}$ appears

to be proportional to $K^{-1/2}$. It might be mentioned that K was also evaluated using the Robinson, Oen, and Holmes¹⁴ neutron spectrum for graphite-moderated reactors for the calculation of $K(\text{fast})$. In this case, the slopes of the lines in Fig. 18.6 are unchanged, but the values of K are increased by a factor of about 3. The dashed curve labeled " $\tau_{1/2} \propto K^{-1/2}$ " in Fig. 18.6 corresponds to the line previously determined from electron and gamma irradiation experiments.^{8,10}

The slopes of the lines in Fig. 18.6 indicate that the mechanism which operates in Cu-Al corresponds to $p = -1/2$ in Table 18.1. Also, the results of Blewitt *et al.*,¹⁹ on Cu-13 at. % Zn suggest that the diffusion-enhancing defect is the vacancy²⁰ and, as we have stated, the previous temperature-dependence experiment on freshly prepared samples, such as those used in the Series I experiments, indicates that the appropriate activation energy, E in Table 18.1, is $\frac{1}{2}E_{mv}$. However, we are not able at this time to explain the longer $\tau_{1/2}$ for the runs of Series II (Fig. 18.6), and it is felt that any further comment on the mechanism of radiation-enhanced diffusion in Cu-Al should await more understanding on this point.

18.3 STRESS RELAXATION IN IRON ALLOYS CONTAINING CARBON AND/OR NITROGEN

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Present knowledge of the effect of radiation damage on iron-carbon and iron-nitrogen alloys is quite limited. It has been shown that neutron irradiation can markedly increase the rate of decomposition of the supersaturated solid solution of carbon in iron.²¹ The exact mechanism by which the carbon is removed from solution in irradiated alloys is uncertain. Since carbon and

¹⁹T. H. Blewitt *et al.*, "Mechanism of Annealing in Neutron Irradiated Metals," p 84 in *Creep and Recovery*, American Society for Metals, Metals Park, Ohio, 1957.

²⁰M. S. Wechsler, "Fundamental Aspects of Radiation Effects on Diffusion-Controlled Reactions in Alloys," Symposium on Radiation Effects on Metals and Neutron Dosimetry, ASTM, Los Angeles, Oct. 1-5, 1962.

²¹H. Wagenblast and A. C. Damask, *Phys. Chem. Solids* 23, 221 (1962).

nitrogen occupy interstitial lattice positions in the solid solution, it is unlikely that the lattice defects produced by irradiation can change the rate of diffusion of these elements in the lattice. Thus the radiation-damage effects observed in iron-carbon alloys are expected to be somewhat different from those observed in substitutional alloys such as copper-aluminum.

If the damping capacity of an iron-carbon specimen vibrating at 1 cps is measured as a function of temperature, a sharp maximum is found at 40°C. The height of this damping peak is proportional to the amount of carbon in solution.²² Carbon atoms associated with other impurity atoms or dislocations or in precipitates do not contribute to this damping peak, but may give rise to damping peaks at other temperatures.^{22,23} In a similar way, the magnitude of stress relaxation (at constant strain) is proportional to the amount of carbon in solid solution. Also, the time constant of the stress relaxation at a given temperature is a measure of the jump frequency of the carbon atoms.

Wagenblast and Damask²¹ made measurements of the damping capacity of iron-carbon wires after irradiating them in the reactor and removing them for measurements. It seems worthwhile to attempt making both stress-relaxation and damping-capacity measurements during reactor irradiation. There are two advantages of making stress-relaxation measurements on these alloys in addition to damping measurements. First, the stress-relaxation measurements can be made at lower temperatures than damping measurements. This is important because carbon and nitrogen diffuse at appreciable rates at room temperature and some of the defects introduced by irradiation diffuse rapidly even below room temperature. Second, it is sometimes possible to separate the relaxation data into several superimposed relaxations. Thus, it has been shown that the relaxation curve for iron-nitrogen alloys can be analyzed in terms of four separate relaxation processes.²⁴ Hence, by stress-relaxation measurements, it may be possible to observe

relaxations due to carbon-vacancy or carbon-interstitial pairs induced by irradiation.

During the past year, equipment has been designed for measuring both stress relaxation and damping capacity in the reactor. The stress-relaxation apparatus has been built and tested. In addition, heat treating equipment for introducing various amounts of carbon and nitrogen into iron has been built. The stress relaxation is detected by measuring the resonant frequency of a stretched flexible wire. For a perfectly flexible string, the resonant frequency is proportional to the square root of the stress. In the present case, the strain on the wire should not exceed 5×10^{-4} , in order to prevent plastic flow. It was calculated that, in order to approach the flexible-string case, the wires should be ≤ 0.005 in. in diameter and 4 in. long. The space available for the apparatus in the reactor facility is quite small. Because of this limited space it seemed that only capacitive methods of driving and detecting vibrations could be used.

Figure 18.7 is a schematic diagram of the specimen, the apparatus used to stress the specimen, and the capacitance plates for detecting and driving oscillations. Three small metal bellows are used to stress the specimen, to lock the specimen at constant strain, and to release the stress. The driving electrode is positioned at the midpoint of the specimen so that, when voltage is applied, the specimen is forced to move in a plane normal to the plane which passes through the two pickup electrodes. The specimen acts as a grounded shield between the two electrodes, and as it moves between them, it changes their capacitance.

Figure 18.8 is a block diagram showing the electronic equipment used in driving the specimen and measuring its resonant frequency. The change in capacitance of the pickup electrodes is detected by a bridge operating at 100 kc. The bridge contains a two-stage amplifier tuned to 100 kc and a diode detector to give a dc voltage which is proportional to the change in capacitance unbalance. The time constant of the detector is short compared with the period of the specimen oscillation. The output of the bridge is further amplified and fed into the driving electrode. A phase shift control is provided so that the phase angle of the driving voltage with respect to the detected signal can be adjusted until resonance occurs. The resonant frequency is then measured

²²J. L. Snoek, *Physica* 8, 711 (1941).

²³L. J. Dijkstra and R. J. Sladek, *Trans. AIME* 5, 69 (1953).

²⁴D. Keefer and C. Wert, *AROD* 2085:4, *AROD* 2085:3.

Fig. 18.7. Schematic Drawing of Stress-Relaxation Apparatus.

Fig. 18.8. Block Diagram of Components for Stress-Relaxation Measurements.

and recorded continuously as the specimen relaxes.

A second apparatus under construction is a flexure pendulum. In this apparatus, Fig. 18.9, a specimen about $\frac{1}{4} \times 1 \times 0.003$ in. is clamped at one end and is vibrated in flexure. A vane, attached to the free end, moves between two capacitance plates. The change in capacitance

induced by the moving vane is detected and recorded. The pendulum is set in motion by applying a voltage pulse between the specimen and a plate parallel to the specimen. The rate of decay of the vibrations so induced gives a measure of the damping capacity. This apparatus will be used to measure the change in the amount of carbon or nitrogen in solid solution as speci-

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Fig. 18.9. Flexure Pendulum.

mens are irradiated in the reactor at slightly above reactor ambient temperatures. For a frequency of 20 cps the nitrogen peak in iron occurs at 60°C. By adjusting the specimen to vibrate at 20 cps and maintaining the specimen at 60°C, the change in the nitrogen peak during irradiation can be observed. This apparatus will also be suitable for electron bombardment experiments, because of the thin specimens which can be used.

18.4 ATOMIC REARRANGEMENTS IN DEFORMED Cu-Si AND Cu-Si-Mn ALLOYS²⁵

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The effect of room temperature deformation and subsequent isochronal annealing on the electrical resistivity, the x-ray diffraction pattern, lattice parameter, stacking fault probability, microhardness, yield stress, and microstructure have been recorded for Cu-6.7 at. % Si (alloy A) and Cu-6.7 at. % Si-1.3 at. % Mn (alloy B). The high stacking fault density produced by deformation is accompanied by a measurable change in the other physical and mechanical properties. Both the stacking faults and residual elastic strains become zero as the temperature is raised to 350°C, but solute clustering (or possibly the formation of an as yet undetected precipitate) reaches a maximum. Between 350 and 550°C, the solute dissolves on increasing the temperature and re-forms on decreasing the temperature. Once recrystallization has occurred, the effect is no longer reversible. The deformation is considered to enhance the redistribution of solute by providing nucleation sites rather than point defects.

²⁵Abstract of paper presented by H. M. Otte at the International Conference on Crystal Lattice Defects, Kyoto, Japan, Sept. 7-12, 1962.

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19. Engineering Properties

19.1 IN-PILE STRESS RUPTURE EXPERIMENTS

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The interest in high-temperature nuclear reactors for power production has created a need for information about the effect of reactor environment on the mechanical properties of structural materials at elevated temperatures, especially when these are selected for such critical applications as fuel element containment. Tests of several alloys have been operated in the poolside facility of the Oak Ridge Research Reactor. The tests were conducted on tubular specimens, internally pressurized to the desired tangential stress level, and maintained at the selected temperature during reactor operation until failure occurred. The times-to-rupture were then compared with the times for similar specimens tested out of the reactor. Postirradiation examination of the specimens included measurements of dimensional changes and metallographic studies.

The recent work includes tests on Inconel, type 304 stainless steel, Zircaloy-2, and niobium-1% zirconium alloy. The irradiation methods for these tests have been previously described.⁴

Inconel

Creep-rupture experiments in the ORR at 1500°F with air atmosphere on two commercial heats of Inconel, heat A (INCO No. NX 8962) and heat B (INCO No. NX 5757), were previously reported.^{4,5}

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³Section Head until July 1, 1962.

⁴W. E. Brundage *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, pp 124-33.

⁵N. E. Hinkle *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1960, ORNL-3017, p 120.

Both heats showed a decreased rupture life due to the irradiation. Also, the deformation at fracture for heat A, as indicated by the increase in diameter, was observed to be smaller for the irradiated than for the control samples. Recently, however, similar measurements for heat-B samples showed the opposite effect, that is, about two times greater deformation at rupture for the in-pile specimens.

To investigate the matter further, an in-pile experiment, again in air and at 1500°F, was operated to determine the amount of deformation of heat A after predetermined creep times at selected stress levels. The results, shown in Table 19.1, indicate no gross effect of the irradiation on the creep rate for this heat.

Postirradiation examination of irradiated specimens from the two heats showed that intergranular separation was localized in heat A (Fig. 19.1) and general in heat B (Fig. 19.2).

Specimens fabricated from six special laboratory heats of Inconel, which contained varying amounts of either natural or isotopically-enriched boron, were examined after the previously reported⁴ stress-rupture tests in the ORR. The tests were performed to determine whether the reduction in the high-temperature stress-rupture strength previously noted⁵⁻⁷ was due to the $B^{10}(n,\alpha)Li^7$ reaction, since Inconel contains 30 to 100 ppm natural boron. These results did not show a clear correlation between the reduction in strength and the increasing B^{10} content.

It now seems unlikely that a confusion of specimen material had occurred, as was at first suspected. Metallographic examination of both the unirradiated and irradiated specimens did, however, present evidence of considerable variation

⁶J. C. Wilson *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1958, ORNL-2614, pp 106-11.

⁷N. E. Hinkle *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1959, ORNL-2829, pp 214-18.

Table 19.1. Effect of Neutron Bombardment on the Creep Rate of Inconel Heat A (INCO NX 8962) in Air at 1500°F

Specimen No.	Stress (psi)	Time (hr)	Maximum Transverse Strain (%)	Rate (%/hr)
Irradiated				
41-3	5000	85	0.6	0.0071
41-7	5000	95, ruptured	1.0	0.0105
Control				
C-279	5000	45	0.4	0.0089
		90	1.1	0.0121
		200, ruptured	7.0	0.0350
C-291	5000	53	0.3	0.0567
		125	1.2	0.0096
		240, ruptured	5.0	0.0208
Irradiated				
41-1	4000	130	0.4	0.0032
41-5	4000	225	0.5	0.0022
41-8	4000	224, ruptured	1.0	0.0045
Control				
C-12	4000	110	0.7	0.0064
		150	1.6	0.0107
		245, ruptured	7.0	0.0286
C-281	4000	115	0.15	0.0013
		280	0.5	0.0018
		630, ruptured	3.0	0.0048
Irradiated ^a				
41-2	3000	330	0.35	0.0106
41-4	3000	530	0.4	0.0008
41-10	3000	890	0.95	0.0011

^aThe control samples for a stress of 3000 psi have not yet been run.

between the different heats in grain size, precipitate concentration, and stringer-type inclusions. A rather large variation in the stress-rupture properties of the materials was also noted in the out-of-reactor tests. These points seem to indicate that the stress rupture strength during irradiation was not directly related to the B¹⁰ con-

tent of the alloy, but was more dependent on other material variables. One complicating factor is that evidence exists which indicates that the boron was not evenly distributed in a given heat. Figure 19.3 is a transverse section through the wall of one specimen. The void (which contained an inclusion lost in the final polishing) appears

Fig. 19.1. Transverse Section of In-Pile Tube-Burst Specimen of Inconel Heat A (INCO No. NX-8962). Note the localized areas of intergranular separation. Test temperature, 1500°F; stress, 5000 psi; rupture time, 91 hr. As polished. 100X. Reduced 23.5%.

Fig. 19.2. Transverse Section of In-Pile Tube-Burst Specimen of Inconel Heat B (INCO No. NX-5757). Note that the intergranular separation is general throughout the wall. Test temperature, 1500°F; stress, 5000 psi; rupture time, 66 hr. As polished. 100X. Reduced 23.5%.

Fig. 19.3. Transverse Section Through the Wall of a Specimen of Inconel OB Tested in the Reactor. Note the change in wall thickness associated with the hole. The void was apparently pressurized during part of the test. 100X. Reduced 23.5%.

to have been pressurized during the test, causing a deformation of the specimen wall. Boron analysis of samples from different portions of the material also indicated that the boron content of the alloys varied considerably. Nonhomogeneity of the boron content in some or all of the alloys could have destroyed the trend expected from the helium production. This, however, should also have caused a large scatter in the tests on irradiated samples, which was not observed.

The transverse deformation at fracture for irradiated Inconel, obtained from measurements of the maximum diameter increase of the irradiated specimens, was much smaller than that of the control specimens. These data are plotted in Fig. 19.4, in which information is also given of the stress-rupture life and the measured boron content.

The fractures were due to the intergranular separation often observed in high-temperature creep, as shown in Fig. 19.5. The fracture is localized, and a pattern of spaced voids along some grain boundaries can be seen. A typical out-of-pile specimen section is shown in Fig. 19.6, in which

more regions of grain boundary separation can be observed. The spaced voids along the grain boundaries were not observed in the control specimens.

Type 304 Stainless Steel

Tube-burst tests were continued on the type 304 stainless steel tubing selected for EGCR fuel-element cladding. The tubing had a diameter of 0.750 in. and a wall thickness of 0.020 in.

The results reported in the previous report⁴ were thought to refer to a single heat of type 304 stainless steel. More recently it was found that two separate heats were included. Consequently, the previous data have been separated according to heats and plotted along with the recent results^{8,9} in Figs. 19.7 and 19.8. The

⁸J. T. Venard, *Gas-Cooled Reactor Program Quart. Progr. Rept. March 31, 1962*, ORNL-3302, p 232.

⁹J. T. Venard, unpublished data.

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Fig. 19.4. Effect of Neutron Bombardment on the Stress Rupture Properties of the Boron-Adjusted Heats of Inconel.

Fig. 19.5. Transverse Section of an In-Pile Tube-Burst Specimen of Inconel 6B11 Showing the Mode of Failure. Note the concentration and shape of the grain-boundary voids. Test temperature, 1500°F; stress, 3000 psi; rupture time, 684 hr. Etchant, aqua regia. 100X. Reduced 23.5%.

Fig. 19.6. Transverse Section of an Out-of-Pile Tube-Burst Specimen of Inconel 6B11 Showing the Type of Intergranular Separation. Note the absence of the fine, intergranular voids seen in Fig. 19.5. Test temperature, 1500°F; stress, 5000 psi; rupture time, 585 hr. Etchant, aqua regia. 100X. Reduced 23.5%.

Fig. 19.7. Effect of Neutron Bombardment on the Stress-to-Rupture of Type 304 Stainless Steel Alloy A. The out-of-reactor curves are taken from ref 8.

Fig. 19.8. Effect of Neutron Bombardment on the Stress-to-Rupture of Type 304 Stainless Steel Alloy B. The out-of-reactor data were taken from ref 9.

rupture strength of heat A is reduced about 20% at 1300 and 1400°F and somewhat less at 1500°F. In contrast, the radiation has little or no effect on the rupture strength of heat B at 1300°F. No control data are available for heat B at 1600°F.

Precipitates were observed in irradiated specimens tested with air as the pressurizing gas. These precipitates, shown by the Widmanstätten structure in Fig. 19.9, are believed to be CrN and CrN₂. No evidence of a precipitate was observed for samples pressurized with helium.

Zircaloy-2

The Zircaloy-2 tube-burst tests, previously described,⁴ were continued. The data indicate that reactor radiation has little, if any, effect on the stress-rupture properties at 700 and 900°F as shown in Fig. 19.10.

Two specimens of Zircaloy-2 were irradiated and tested in the reactor in order to investigate the effect of neutron bombardment on a strain-aging phenomenon observed¹⁰ in this alloy. One specimen, strain-aged under irradiation at 500°F at 40,000 psi for 860 hr, ruptured at 51,600 psi when the stress was subsequently raised.

The second specimen was strain-aged and irradiated at the same stress for 1500 hr and ruptured when the stress was increased to 55,500 psi. These rupture stresses are more than 25% greater than the corresponding value of 43,000 psi observed¹¹ without strain aging and irradiation. Thus, these results indicate an increase in rupture strength with time of strain aging and irradiation at 500°F and 40,000 psi. Since duplicate out-of-reactor experiments at 500°F, using the same aging stress and time, have not yet been conducted, it is not yet possible to specify the effect of irradiation on the strain-aging phenomenon.

Niobium-1% Zirconium

It was previously reported⁴ that the rupture life of niobium-1% zirconium samples, stressed at 2000°F, was reduced upon irradiation. Recent hot-cell examination has shown, however, that this conclusion was incorrect, in that the failure occurred in the brazed joint rather than in the specimen. Therefore, the reported data represent only a limit for any radiation-induced weakening of this alloy at 2000°F.

¹⁰J. J. Holmes, *The Activation Energies for Creep of Zircaloy-2*, HW-70151 (March 1962).

¹¹C. R. Kennedy, *Metals and Ceramics Div. Ann. Progr. Rept. May 31, 1962*, ORNL-3313, p 45.

Fig. 19.9. Transverse Section of an Irradiated Tube-Burst Specimen of Type 304 Stainless Steel Alloy B Tested at 1600°F and Ruptured After 1430 hr at 2200 psi. Etchant, aqua regia. 200X. Reduced 23.5%.

Fig. 19.10. Effect of Neutron Bombardment on the Stress-to-Rupture of Zircaloy-2 at 700 and 900°F in Helium.

Vacuum fusion analyses on samples of two irradiated specimens stressed at 1800°F indicated slight increases in the concentrations of hydrogen, nitrogen, and oxygen. This represents reasonably good atmosphere control, and the increase in the amount of impurities is not believed to have a significant effect on the high-temperature stress-rupture properties.

Neutron Flux Monitoring

Neutron flux monitors have been included in each assembly of tube-burst specimens irradiated in the ORR poolside. Monitors of cobalt, sulfur, Np^{237} , and U^{238} were irradiated during the entire time of the test. The disintegration rates of the neutron-reaction products were determined after

removal from the reactor, and the average neutron flux was calculated.

The results of the neutron flux monitoring tests in the ORR poolside facility positions P-5 and P-6 are listed in Table 19.2. The ranges of values listed for each entry are probably due to such factors as the variations in the fuel loading of the reactor and the relative position of the monitors in the experiment cans.

A source of error in the use of the $\text{Np}^{237}(n, \text{fission})$ reaction as a threshold monitor has been found. The $\text{Np}^{237}(n, \gamma)\text{Np}^{238}$ reaction for thermal neutrons, leading to a buildup of Np^{238} and Pu^{238} which yield fission by thermal neutrons, had been overlooked. For short irradiations or low flux the error introduced is negligible. However, for irradiation times exceeding a few days at a flux of 10^{13} neutrons $\text{cm}^{-2} \text{sec}^{-1}$ or greater, the fission products produced by thermal-neutron reactions cause an appreciable error in the calculated fast flux. Since this error is very large for the conditions encountered in these monitoring experiments, the flux measurements with Np^{237} are not considered reliable. The neutron flux determinations using U^{238} are also considered unreliable, possibly because of the U^{235} impurity.

In the future, the Np^{237} and U^{238} monitors will be shielded with cadmium to reduce the effect of the thermal flux. Cadmium-shielded cobalt monitors will also be added, to enable the separate determination of the thermal and epithermal fluxes.

Table 19.2. Neutron Flux Measurements in the ORR Poolside Facility Positions P-5 and P-6

Monitor	Energy Range	Flux (neutrons $\text{cm}^{-2} \text{sec}^{-1}$)	
		P-5	P-6
		$\times 10^{13}$	$\times 10^{13}$
Cobalt	Thermal	3-7	5-8
Np^{237}	> 0.6 Mev	3-9	10-35
U^{238}	> 1.5 Mev	1.5-15	20-50
S^{32}	> 2.3 Mev	0.1-0.5	0.7-1.3

Discussion

The occurrence of spherical voids arranged along grain boundaries and separated grains with "scaloped" edges in irradiated Inconel specimens was first thought to be due to helium production from the fissioning of the B^{10} impurity.⁷ The helium, precipitating out at grain boundaries, would act to increase the local stress due to the internal pressure and to form stress-raisers by reducing the area of the grain boundary. This offers a possible explanation of the observed reduction in rupture strength. To test this hypothesis, samples from special heats of Inconel, containing varying amounts of B^{10} and B^{11} , were tested in the reactor. The expected correlation between reduced rupture strength and the B^{10} concentration was not observed, although the B^{10} concentration was varied by a factor of about 100.

The lack of supporting evidence of the effect of boron for the Inconel probably rules out this effect in the stainless steel, since the concentration of boron in the stainless steel was lower by a factor of 5 than in the Inconel.

Another point of interest is that the reduction in the rupture strength of these materials during irradiation is accompanied by a decrease in the deformation at rupture in the stainless steel and all but one of the heats of Inconel.

The high-temperature property changes induced by irradiation appear to be connected with an increased grain-boundary-void nucleation or growth. Several models have been proposed to explain voids formed in grain boundaries transverse to the major stress direction during creep. One of the mechanisms proposed by Greenwood *et al.*¹² is that the voids grow as a result of the condensation of vacancies generated during creep. Also, Balluffi and Seigle¹³ postulate that voids, nucleated at grain boundaries, grow by migration of vacancies from the boundaries into the voids. If the precipitation of vacancies is important in the growth of intergranular voids, the dynamic vacancy supersaturation existing during neutron bombardment would accelerate the void

¹²J. N. Greenwood, D. R. Miller, and J. W. Suiter, *Acta Met.* 2, 250 (1954).

¹³R. W. Balluffi and L. L. Seigle, *Acta Met.* 5, 449 (1957).

growth and permit failure to occur sooner and at lower strains than in the absence of radiation.

The intergranular cavitation was not observed in the tests on Zircaloy-2 and the niobium-1% zirconium alloy. This was probably due to the fact that these alloys were not investigated in the high-temperature creep range.

It is unlikely that the mechanisms of the void formation and growth will be determined by testing commercial alloys. The mechanisms are probably very dependent upon such material variables as impurities, precipitates, grain size, and relative grain orientation. Sufficient control over these parameters is not easily obtained in these alloys.

19.2 IMPACT TESTS ON IRRADIATED PRESSURE-VESSEL STEELS

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Surveillance samples of ASTM A212, Grade B, steel from the pressure vessel of the SM-1 reactor

¹⁴Reactor Division,

at Fort Belvoir, Virginia, were placed in the reactor at the initial startup in April 1957. Eight capsules containing impact bars were located on the core support plate 15.0 in. from the vertical center line of the reactor, with the bottom ends of the impact bars 15.0 in. from the core center. Each capsule contained three subsize Izod impact specimens with six notches each. Since there is a steep fast-neutron flux gradient at this position, each notch was exposed to a different fast-neutron flux. The exposure temperature of the capsules was about that of the exit coolant (450°F).

Thus far, two capsules have been removed; the first after 16.4 Mwyr of operation and the second after 20.8 Mwyr. The impact tests were conducted at the Oak Ridge National Laboratory in Hot Cell 4 of Building 3025. Unirradiated specimens from the same steel plate were tested in the hot cell along with the irradiated surveillance specimens. The results of the impact tests are shown in Figs. 19.11 and 19.12. Since only three tests were possible for each notch location and exposure, the impact curves are not very well defined. Therefore, an estimate of an effective brittle-ductile transition temperature is rather

Fig. 19.11. Impact Energy vs Test Temperature for Surveillance Specimens of ASTM A212, Grade B Steel from SM-1 Pressure-Vessel Material After 16.4 Mwyr of Reactor Operation.

Fig. 19.12. Impact Energy vs Test Temperature for Surveillance Specimens of ASTM A212, Grade B Steel from SM-1 Pressure-Vessel Material After 20.8 Mwyr of Reactor Operation.

difficult. However, the following procedure was adopted. A transparent overlay was constructed, which defined the scatter-band for the unirradiated samples. The overlay was then translated along the temperature axis so that the three points for a given notch were best fitted by eye inside of the scatter band. The amount of translation (in °F) thus determined was taken to be the increase in transition temperature. The results of this analysis for the two capsules are given in Table 19.3.

It is current practice, particularly as concerns design and operating criteria for reactor pressure vessels, to use the Charpy V-notch impact sample in the determination of transition temperatures. It has been found¹⁵ that the irradiation-induced increase in transition temperature for the Charpy V-notch sample is roughly 25°F higher than for the subsize Izod sample. On this basis, the corresponding Charpy V-notch transition temperature increases are also given in Table 19.3.

¹⁵R. G. Berggren, *USAEC Conference on the Status of Radiation Effects Research on Structural Materials and the Implications to Reactor Design*, TID-7588, p 91 (October 1960).

A study of the neutron flux distribution has been reported¹⁶ for a mockup of the SM-1, containing mockups of the thermal shield and pressure vessel. The neutron flux measured with sulfur detectors was found to be 4.8×10^2 neutrons $\text{cm}^{-2} \text{sec}^{-1} \text{w}^{-1}$ at the pressure vessel and 13.9×10^2 neutrons $\text{cm}^{-2} \text{sec}^{-1} \text{w}^{-1}$ at a position corresponding to notch 5 of the surveillance samples. Thus, for notch 5 of capsule I (16.4 Mwyr) and capsule II (20.8 Mwyr), we calculate sulfur doses of 6.9×10^{17} and 8.7×10^{17} neutrons/ cm^2 , respectively. A formula relating transition temperature increase, ΔTT , and neutron dose has recently been given by Nichols and Harries¹⁷

$$\Delta TT = 49 \Phi(\text{Ni})^{1/2}, \quad (1)$$

¹⁶K. C. Sontheimer *et al.*, *Fast Neutron Flux Measurements and Analysis in SM-1 and PM-2A Core and Vessel Mock-ups*, APAC-95 (March 31, 1962).

¹⁷R. W. Nichols and D. R. Harries, "Brittle Fracture and Irradiation Effects in Ferritic Pressure Vessel Steels," Symposium on Radiation Effects on Metals and Neutron Dosimetry, ASTM, Los Angeles, October 1-5, 1962.

Table 19.3. Increase in Brittle-Ductile Transition Temperature for the SM-1 Surveillance Samples

Notch Number ^a	Subsize Izod Transition Temperature Increase (°F)	
	[Charpy V ΔTT Values ^b in Parentheses]	
	Capsule I 16.4 Mwyr	Capsule II 20.8 Mwyr
1	45 (70)	51 (76)
2	58 (83)	67 (92)
3	69 (94)	76 (101)
4	88 (113)	109 (134)
5	120 (145)	141 (166)
6	130 (155)	150 (175)

^aNotches numbered in order of decreasing height (decreasing distance from horizontal core midplane).

^bCalculated according to ΔTT (Charpy V) = ΔTT (Subsize Izod) + 25°F. (See ref 15.)

where $\Phi(\text{Ni})$ is integrated flux obtained with a nickel threshold detector (threshold energy, 2.9 Mev) in units of 10^{18} neutrons/cm². The sulfur flux should be about the same as for nickel, since the threshold energy for sulfur is 3.0 Mev. Equation (1) was determined for Charpy V-notch samples irradiated at 300°F at much higher fluxes than the SM-1 surveillance samples. If now we substitute into Eq. (1) the integrated fluxes, 6.9×10^{17} and 8.7×10^{17} neutrons/cm² given above for the surveillance samples, we find that the predicted ΔT values are 40 and 45°F for notch 5 of the capsules I and II, respectively, as compared to the values 145 and 166°F (Table 19.3) obtained from our impact tests on the surveillance samples.

The discrepancy between the transition temperature increases observed for the surveillance samples (145 and 166°F) and those predicted on the basis of Eq. (1) (40 and 45°F) may be due to the following reasons:

1. Lack of precision in the impact tests.
2. Errors in neutron dosimetry.
3. Differences in neutron spectrum.

4. The higher exposure temperature of the surveillance specimens (450°F) as compared with that of the samples on which Eq. (1) is based (300°F). This may be a factor, since some steels have shown a maximum between 300 and 500°F in the curve relating embrittlement to irradiation temperature.
5. The higher flux, by a factor of about 10, for the accelerated-irradiation samples as compared with that for the surveillance samples. It has been pointed out^{18,19} that, if the changes in properties of pressure vessel steels upon irradiation are associated with radiation-enhanced diffusion, then larger effects are to be expected in certain cases when a given dose is received at a lower flux.

¹⁸M. S. Wechsler and R. G. Berggren, "The Radiation Embrittlement of Reactor Pressure Vessels. I. Fracture and Radiation Damage," to be published.

¹⁹M. S. Wechsler, "Fundamental Aspects of Radiation Effects on Diffusion-Controlled Reactions in Alloys," Symposium on Radiation Effects on Metals and Neutron Dosimetry, ASTM, Los Angeles, October 1-5, 1962.

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Part VI. Reactor Materials

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20. Fuel Materials

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20.1 POSTIRRADIATION EXAMINATION OF GRAPHITE MATRIX FUEL

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MTR-Irradiated Fueled Graphite

Examination of the six MTR-irradiated fueled graphite experiments is nearly complete. Results from the first three experiments in this series were reported previously.^{2,3} As shown in Table 20.1, three different types of fuel were used. Experiment MTR-48-4 contained four fuel pellets sealed in a low-permeability graphite can, while experiments -5 and -6 contained single fuel pellets and were unsealed. All elements were purged with a slow flow of helium, which facilitated fission-gas release measurements during irradiation.

¹Metals and Ceramics Division.

²O. Sisman *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1960, ORNL-3017, pp 113-14.

³O. Sisman *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, pp 110-11.

The irradiation effects on experiment -4 were similar to those of previous experiments. There were small decreases in fuel-pellet dimensions, both length and diameter, for experiments -4 and -5. No dimensional changes were apparent for the -6 fuel pellet, which contained coated (U,Th)C₂ particles. The appearances of the three types of fuel pellets are compared in Fig. 20.1. The -4 fuel pellets, which contained uncoated fuel particles, were coated with pyrolytic carbon. With the exception of two small cracks in the -5 fuel pellet (one of which is apparent in Fig. 20.1b), there were no changes in appearance caused by the irradiation.

Samples of the fuel pellets and graphite cans were analyzed to determine the extent of uranium and fission-product migration. As was the case in earlier experiments, significant migration was detected, and is illustrated in Fig. 20.2. The original uranium concentration in the -5 fuel pellet was 0.0904 g of U per cm³, or 2.3×10^{17} atoms of U per mg. The uranium concentration in the graphite can after irradiation ranged from about 4% of that in the fuel near the inside surface to

Table 20.1. Data Summary for MTR-48 Experiments

Experiment No.	Type of Fuel Particle in Graphite Matrix	Fuel Loading (g of U ²³⁵ per cm ³)	Maximum Fuel Temperature (°F)	Burnup (at. % U ²³⁵)
MTR-48-4	Uncoated UC ₂	0.169 to 0.517	2500 to 2800	3.5 to 0.9
MTR-48-5	Pyrolytic-carbon-coated UC ₂	0.0841	2600	17
MTR-48-6	Pyrolytic-carbon-coated (U,Th)C ₂	0.088	2000	^a

^aAnalysis incomplete, probably ~2.3.

Fig. 20.1. Appearance of Irradiated Fuel Pellets from MTR-48 Experiments. Reduced 36%.

Fig. 20.2. Distribution of Uranium and Fission Products Across A Graphite-Can Wall, MTR-48-5.

about 0.4% near the outside surface. The fission-product concentrations found in the can represent a significant fraction of the total formed — about 30% for Cs¹³⁷.

Metallographic examination of the fuel pellets indicated that the structure of the uncoated fuel particles was sensitive to uranium burnup. Fuel particles from pellets A and D, experiment -4, are shown in Fig. 20.3. There was no visible change in the structure of particles from pellet D (0.85 at. % U²³⁵ burnup), but particles from pellet A (3.5 at. % U²³⁵ burnup) had undergone considerable change. These particles had become porous and amorphous in appearance, and part of the fuel had migrated into the surrounding graphite. The central temperatures were about 2500 and 2800°F for fuel pellets D and A respectively.

All the fuel-particle coatings observed during the examination of the -5 fuel pellet were broken. There were no differences in the appearances of particles from different areas, that is, the center of the fuel pellet as compared with regions near the outside surface. As shown in Fig. 20.4, the fuel particles had lost their well-defined microstructure.

ORR-B9-6 Experiment

Postirradiation examination has been completed on capsule B9-6, which contained pyrolytic-carbon-coated uranium carbide particles dispersed in a graphite matrix. The graphite body was coated with siliconized SiC. This specimen was irradiated at 2000°F for 362 hr to a burnup of 3000 Mw per metric ton of uranium. Postirradiation examination revealed that the capsule had broken circumferentially about $\frac{1}{8}$ in. from the top, and the top section was in several wedge-shaped pieces. The center of the specimen was a loose mixture of coated particles and small graphite chunks. When

Fig. 20.3. Representative Fuel Particles from MTR-48-4, Illustrating Microstructural Differences Associated with Different Burnup Levels. Etched. 250X. (a) Particle from fuel pellet D, 0.85 at. % U^{235} burnup. Similar to unirradiated particle. (b) Particle from fuel pellet A, 3.5 at. % U^{235} burnup. Note porosity in fuel particle. Reduced 15%.

Fig. 20.4. Representative Fuel Particles from MTR-48-5. Note fractures in pyrolytic-carbon coatings and absence of a well-defined microstructure in uranium carbide particles. Etched. 150X. Reduced 16%.

the loose material was removed, a cavity roughly $\frac{3}{8}$ in. in diameter extended to within about $\frac{1}{8}$ in. of the bottom of the specimen. Pieces of the top section and loose particles that were removed from the cavity are shown in Fig. 20.5, and the cavity is shown in Fig. 20.6. Since air leaked into the helium sweep system during part of the irradiation period, it is thought that the cavity may have been caused by selective oxidation in the central and hottest part of the specimen.

Most of the coated particles removed from the cavity had retained their smooth surface, but about 3 to 5% of them were broken. Several particles were covered with bits of silicon from the graphite-body coating. Smooth and silicon-flecked particles removed from the specimen center are shown in Fig. 20.7. Typical unbroken particles are shown in Figs. 20.8 and 20.9. All unbroken particles examined showed migration of the uranium carbide outward into the laminar coating. Bits of the carbide can be seen in the particle coating shown in Fig. 20.8. The migration did not extend completely through the coating, and thus it appears that migration does not occur at the temperature of the outer part of the coating. It is possible, but perhaps less likely, that the distance of the migration is a function of time.

An unirradiated particle from the same batch is shown in Fig. 20.10. This particle shows no

Fig. 20.5. Broken Top Pieces of Irradiated Capsule B9-6 and Pyrolytic-Carbon-Coated Uranium Carbide Particles Removed from Capsule Center. 2.2X.

Fig. 20.6. Irradiated Capsule B9-6, Showing Cavity That Remained After Removal of Material Shown in Fig. 20.5. 2X.

Fig. 20.7. Pyrolytic-Carbon-Coated Uranium Carbide Particles from Capsule B9-6. Some particles are flecked with silicon. 20X.

migration of uranium carbide. It may be noted, however, that the siliconized SiC penetrated the graphite specimen body and surrounded the coated particle.

Metallographic examination of broken sections showed no relation between cracks and fuel-migration areas. A typical broken particle is

shown in Fig. 20.11. The cracks may have passed through these areas at other places, but were not connected at any of the sectioned planes. Metallographic examination of the fuel particle revealed an integral grain structure; however, an unidentified second phase was observed in some of the coated particles. The second phase existed mainly in the grain boundaries.

Fig. 20.8. Double-Centered Pyrolytic-Carbon-Coated Uranium Carbide Particle from Irradiated Capsule B9-6, Showing Bits of Uranium Carbide That Have Migrated into the Coating. 250X.

Fig. 20.9. Typical Unbroken Particle from Irradiated Capsule B9-6, Showing Uranium Carbide Migration Outward Through Part of the Pyrolytic-Carbon Coating. 250X.

Fig. 20.10. Unirradiated Pyrolytic-Carbon-Coated Particle from the Same Batch as Capsule B9-6 Particles. Siliconized SiC penetrated the graphite body and surrounded the particle. 250X.

Fig. 20.11. Typical Broken Pyrolytic-Carbon-Coated Particle from Irradiated Capsule B9-6. 250X.

20.2 COATED FUEL PARTICLES

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Postirradiation Examination of Capsules LCP-1 and LCP-2

Uranium carbide fuel particles coated with pyrolytic carbon were irradiated in air-cooled core facilities in the LITR.⁴ Two types of coatings were tested: capsule LCP-1 contained particles with a columnar coating, supplied by High Temperature Materials, Inc., and capsule LCP-2 contained particles with a laminar coating supplied by the 3M Company. Both types of coatings were approximately $82\ \mu$ thick. Each capsule consisted of two separate sections, denoted "a" (top) and "b" (bottom) as shown in Fig. 20.12. The fuel particles were contained in tantalum cans, which were sealed inside and insulated from the Inconel irradiation capsules. The capsule fill gas was purified helium.

The capsules were first pierced for fission-gas analysis. The results, along with irradiation conditions, are shown in Table 20.2. The low fission-gas release values for both "a" sections, compared with the "b" sections, indicate leakage. The appearance of the internal components from the two "a" sections, especially the tantalum-clad center thermocouple, was quite different. There was no obvious oxidation of the tantalum in LCP-1a, indicating that leakage in this capsule occurred after the irradiation. Conversely, the interior of LCP-2a was badly oxidized, indicating leakage during irradiation, that is, at elevated temperature. There was no evidence of oxidation in either LCP-1b or LCP-2b.

The appearance of the fuel particles had not changed markedly during irradiation. A few particles with cracked coatings were found in all four sections. Most of the HTM-type coatings which cracked were those on "doublet" particles, illustrated in Fig. 20.13. The particles from LCP-2a were duller in appearance and exhibited more cracked coatings than those from LCP-2b, probably due to oxidation of the carbon coatings in the former. In addition, as shown in Table

20.2, acid leaching of batches of the fuel particles indicated that a larger fraction of the coatings failed in the "a" sections of the capsules.

Samples of the fuel particles from both sections of each capsule were examined metallographically. Most of the coatings showed some damage

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Fig. 20.12. Diagram of Coated-Particle Irradiation Capsule.

⁴The irradiations were performed by the Reactor Division.

Table 20.2. Results of Examination of Irradiated Coated Particles

Capsule No.	Central Temperature (°F)	Uranium Burnup		Kr ⁸⁵ Fission Gas Released ^a (%)	Uranium Removed by Leaching ^b (%)
		Mwd/metric ton	At. %		
LCP-1a	1950	45,000	4.7	0.01	22
LCP-1b	2100	45,000	4.7	0.1	2.0
LCP-2a	2400 ^c	70,000 ^d	7	^e	10
LCP-2b	2300 ^f	70,000 ^d	7	0.2	0.62

^aExpressed as percentage of that produced.

^bExpressed as percentage of total uranium.

^cThermocouple indicated a temperature rise to 3200°F and then failed.

^dEstimated.

^eKrypton-85 was detected but could not be counted; less than 0.1% of Xe¹³³ was released.

^fThermocouple failed two days after startup; temperature was probably higher than in capsule LCP-2a.

resulting from the irradiation. Examples of these effects on the HTM coatings (LCP-1) are shown in Figs. 20.13 and 20.14. There appeared to have been some reaction at the core-to-coating interfaces; however, most of the detail in these regions was lost during sample preparation due to the loose nature of the material.

The coated particles from LCP-2 were somewhat angular, rather than spherical, as illustrated in Fig. 20.15. The effect of oxidation is illustrated by Fig. 20.16, which shows typical particles from LCP-2a and LCP-2b. This figure also shows evidence of some reaction at the core-to-coating interfaces. The pyrolytic carbon in this region appeared to be less dense, and numerous "spear-head" fractures had begun to penetrate the coating. A typical fractured particle is shown in Fig. 20.17. The fuel cores in both types of particles had changed in appearance, and in most cases the microstructure was less well defined after irradiation. Fine porosity and a small amount of second-phase material were observed in the fuel cores from LCP-2.

Instantaneous Fission-Gas Release

In-pile tests have continued in the ORR B9 and C1 facilities to determine fission-gas release from pyrolytic-carbon-coated uranium carbide particles. These particles are unsupported; that is, they are not contained in a graphite matrix, but are held in

a cylindrically shaped monolayer in a graphite holder. The holder, shown in Fig. 20.18, is vented to allow fission gas released from the particles to be swept from the capsule in a helium stream.

Fission-gas release rates from three basically different types of pyrolytic carbon coatings are being studied. Also, the release rate from bare uranium carbide particles was measured to determine how much of the fission-gas retention could be attributed to a coating. The three coating types are laminar, columnar, and duplex; the duplex coating consists of a laminar inner layer and a columnar outer coating. Sectioned particles of the three coating types and bare uranium carbide are shown in Fig. 20.19. Particle size, coating thickness, manufacturer, and fuel loading are given in Table 20.3.

Experiment B9-7. — The B9-7 capsule, containing HTM-1 columnar-type coated particles, was irradiated for 58 days. The particles were held at 1500°F in a thermal-neutron flux of 3.7×10^{13} *nv* for 43 days. The average fractional release of fission gas during this period is shown in Fig. 20.20. The temperature was subsequently increased to 1650°F by inserting the capsule into a higher-flux region of 5.5×10^{13} *nv*, and irradiation was continued for 15 days.

The fractional release rates of Xe¹³³, Xe¹³⁵, Kr^{85m}, Kr⁸⁷, and Kr⁸⁸ were found to be temperature dependent. When the temperature was decreased from 1500°F to 1370°F with the neutron

Fig. 20.13. Pyrolytic-Carbon-Coated Uranium Carbide Particles from Batch HTM-1. (a) Unirradiated "doublet." As polished. Polarized light. 150X. (b) Broken "doublet" from section "b" of LITR capsule LCP-1. Etched. Polarized light. 150X.

Fig. 20.14. Typical Coated Particles from Batch HTM-1. (a) Unirradiated. Etched. 75X. (b) Irradiated. Etched. 150X. Note basal-plane fractures and reaction area at core-to-coating interface.

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Fig. 20.15. Typical Cross Section of Unirradiated Particle from Lot 3M-SP-2. Used in LITR capsule LCP-2. As polished. 250X.

flux remaining constant, the fractional release rates for all the isotopes, except Xe^{133} , decreased by a factor of approximately 3; the release rate for Xe^{133} decreased by a factor of 5. A total of 12.1% uranium burnup was attained.

Experiment C1-7. — The 3M-SP-2 laminar-type coated particles were irradiated in capsule C1-7 at 1550°F for 33 days at a thermal-neutron flux of $3.1 \times 10^{13} \text{ nv}$. The specimen released fission gas as soon as it was inserted into the reactor. The gas release rate slowly increased until the isotopes generated within the fuel reached a production-decay equilibrium after about 14 days of irradiation. The release rates were reasonably constant for the remainder of the 33-day irradiation period. The capsule was then rapidly cooled by withdrawing it from the reactor flux, and the fission-gas release was measured during the cooling

period. The steady-state fractional gas release after 33 days of irradiation is given in Fig. 20.20.

The steady-state fission-gas release rates and the fission-gas release rates during the cooling period are plotted in Fig. 20.21. The release rates are given in atoms per second for the isotope emerging from the graphite container. The first two points show the steady-state release. As the specimen cooled, the fission-gas release decreased abruptly until the krypton release was no longer detectable. This was caused by a decrease in the gas pressure in the graphite holder when it cooled, which allowed gas to flow into the holder until the pressure was equalized with the external gas pressure. Xenon was always detectable because a large portion of the total xenon is born from iodine that has escaped from the capsule and deposited on a cooler portion of the sweep system. The

Fig. 20.16. Typical Irradiated Particles from Capsule LCP-2. As polished. 250X. (a) Particle from LCP-2a showing oxidation of the coating. (b) Particle from LCP-2b, no oxidation. Reduced 31%.

krypton isotopes have no long-lived precursors; so their total emission is from the fuel particle. The minimum in the xenon curve probably represents the steady-state release rate of iodine from the particles. These rates indicate release of 25% of the total Xe^{133} and 82% of the total Xe^{135} from the coated particle in the form of iodine.

After the cooling dip, shown in Fig. 20.21, the fission gas surged out of the graphite holder. The amount of Xe^{133} released in this surge demonstrated that stored gas was released from within the particle. Stored gas contains relatively large amounts of long-lived gases. The graphite holder would tend to adsorb greater amounts of xenon as it cooled, and thus the graphite could not be the source of the burst. This burst of stored gas is also noticeable in the krypton release, where the outward surge of Kr^{85m} was considerably greater than that of Kr^{87} although the steady-state release of 78-min Kr^{87} was approximately equal to that of 4.4-hr Kr^{85m} .

After the cooling burst, the fission-gas release for all isotopes slowly declined. Xenon release from deposited iodine is natural under these conditions, but krypton release usually terminates at once. This effect is probably caused by fission gas diffusing through the spongy layer between the particle and its coating until the gas reaches a break in the coating. The spongy layer is the dark inner ring shown on 3M-SP-2 in Fig. 20.19.

When the specimen was reinserted into the reactor after the cooling burst, the fission-gas release rates were erratic and, in general, higher than for the first month of irradiation. The specimen temperature was then lowered while at constant power so that the temperature dependence of the gas release could be studied. No temperature dependence was observed over a temperature range of 670°F .

The specimen was then irradiated at an increased flux of 5.2×10^{13} thermal neutrons $\text{cm}^{-2} \text{sec}^{-1}$. Fission-gas release continued to be erratic,

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Fig. 20.17. Fractured Particle from Capsule LCP-2b. Etched. 250X.

especially the Xe^{133} release, which fluctuated by large amounts for no apparent cause. For the next 30 days the capsule was irradiated at 1815°F with little effect on the steady-state fission-gas release. There were no significant changes in fractional fission-gas release rates as a function of power level, burnup, or temperature.

When capsule C1-7 was being prepared for irradiation at the higher flux of 5.2×10^{13} thermal neutrons $\text{cm}^{-2} \text{sec}^{-1}$, the temperature of the specimen was increased to 1700°F for about 30

min. This resulted in a burst of activity that was two orders of magnitude higher than any previous release. The gamma-emission spectrum of the fission gas appeared normal in every respect except for the magnitude. Since a burst of stored gas would be rich in long-lived isotopes, it appears that the gas released in this burst was the result of accelerated diffusion. Later the capsule temperature was raised to 1815°F , but no abnormal release rates were noted. The capsule had been irradiated for about a month at 1550°F , and the

Fig. 20.18. Coated-Particle Holder for ORR C-1 and B-9 Facilities.

burst was produced when the temperature was first increased to 1700°F. This burst did not signify fuel damage since no significant change of steady-state fission-gas release resulted. Subsequent attempts to duplicate the heating burst were not successful.

Experiment B9-8. — The B9-8 capsule, containing coated particles of the duplex type from batch NCC-AD, was irradiated for 88 days to a 30% uranium burnup. A sectioned duplex-type particle is shown as NCC-AD in Fig. 20.19. The average fractional fission-gas release rates, at temperatures of 600 to 1700°F, are given in Fig. 20.22. The fission-gas release rate during the first six days was below the detectable range. On the seventh day the release rate increased, reached a steady-state condition after a few days, and remained steady for the 1500°F irradiation period. The average fractional release rates at 1500°F are compared in Fig. 20.20 with those from other tests.

After a 20% burnup had been reached, activity bursts were observed. These bursts continued at irregular intervals throughout the remainder of the 1700°F irradiation period, but did not appear after the temperature was reduced to 800 or to 600°F. The fission-gas release rates showed a marked dependence on temperature during the first few weeks of irradiation, but this dependence decreased as burnup increased. This means that fuel was within the recoil range of the outer particle surface — probably due to both broken coatings and fuel migration into the coating.

Experiment C1-8. — The C1-8 capsule, containing bare uranium carbide particles from batch 3M-101-U, was irradiated for 41 days at 1500°F and at 4×10^{13} *nv* to a 4.3% uranium burnup. While at constant flux, the fission-gas release rates remained unchanged after the first seven days of irradiation. Average fractional release rates are given in Fig. 20.20. No temperature dependence for fission-gas release was found for temperatures up to 1500°F.

The bare uranium carbide particles actually had a layer of porous graphite of about 15- μ thickness (see Fig. 20.19). The bare particles did not release the sudden bursts during steady-state irradiation that were noted from B9-7, B9-8, and C1-7 capsules, strengthening the argument that the bursts indicate cracking of the coatings. The bare particles do release heating bursts during temperature increases. Contrasted to the laminar-coated particles in the C1-7 specimen, the bare particles did not release bursts while cooling. This implies that the cooling burst was caused by a cracking or contraction of the coating.

Activity Bursts Not Due to Temperature Change.

— Activity bursts were noted during the irradiation of all three types of coated particles, but not from the bare uranium carbide particles. These bursts occurred while power level and temperature were at a steady state and at irregular intervals. As shown in Fig. 20.23, the steady-state activity level increased for a few hours and then gradually returned to the previous level. A gas sample taken during a burst was abnormally rich in 5-day Xe¹³³, indicating the release of trapped gas.

It appears that activity bursts are caused by the coating rupture of a single particle. The bursts contain about 1.2×10^{-5} curie of gas, whereas after 30 days of irradiation time a coated particle contains about 1.1×10^{-4} curie of gas. Irradiation of bare particles demonstrated that most of

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3M-SP-2

HTM-1

NCC-AD

UNCOATED URANIUM CARBIDE

Fig. 20.19. Pyrolytic-Carbon-Coated Uranium Carbide Particles. 100X.

Table 20.3. Three Basically Different Types of Pyrolytic-Carbon Coatings

Capsule No.	Coating Type	Manufacturer and Batch No.	Particle Size (μ)		U^{235} (g)
			Average Overall Diameter	Coating Thickness	
C1-7	Laminar	3M Co. 3M-SP-2	414	82	0.087
B9-7	Columnar	HTM, Inc. HTM-1	364	82	0.063
B9-8	Duplex	National Carbon NCC-AD	460	80	0.086
C1-8	Bare	3M Co. 3M-101-U	228	No coating	0.0045

Fig. 20.20. Average Fractional Fission-Gas Release, R/B at 1500°F.

Fig. 20.21. Fission-Gas Release Rates of Batch from 3M-SP-2 Coated Particle During Steady-State Irradiation, Reactor Scram, and Cooling of Capsule CI-7.

Fig. 20.22. Fractional Fission-Gas Release Rates from Pyrolytic-Carbon-Coated Uranium Carbide. B9-8 MCC-AD Duplex.

Fig. 20.23. Typical Activity Burst During Irradiation of Pyrolytic-Carbon-Coated UC_2 Particles.

the generated gas is held in the uranium carbide at temperatures up to 1500°F. If a particle should suddenly release one-tenth of its gas, this would cause the observed burst.

Coating Efficiency. — In evaluating coatings, the efficiency of a coating could be expressed as a relationship between the fractional release rate from coated fuel particles compared with the fractional release rate from uncoated fuel particles. This relationship is given in Table 20.4 as an efficiency factor. It should be noted that this factor for each isotope from a given coating is in close agreement except for the 3M Company laminar coating. This is because the so-called bare particles have a thin, spongy graphite coating that may act as a recoil barrier, while on a broken 3M coating this thin barrier was probably removed along with the laminar shell.

Conclusions. — Results indicate that the failure of the laminar coatings (3M Company) and the columnar coatings (High Temperature Materials, Inc.) occurred as soon as fission heating began. These failures were probably due to differential thermal expansion between the particle and coating, and only a very small percentage of the particles were broken in this way. The NCC duplex coating did not begin to break until 20% burnup had been attained. The small fission-gas release rates up to this burnup were by diffusion through intact coatings.

The particles with the 3M laminar coatings appeared to release fission gas with less holdup than the particles with the HTM columnar or the NCC duplex coatings. This was determined by the relatively larger proportion of long-lived fission gas in the steady-state emission from the HTM and NCC coated particles. Also, the gas release from the 3M coated particles was not temperature dependent, whereas the release from the HTM and NCC coated particles was highly temperature dependent. From these observations it is concluded that the fission-gas release from the 3M coated particles was primarily by recoil from broken and peeled particles and that the initial release from the HTM and NCC particles was primarily by diffusion through the coating.

Table 20.4. Efficiency Factors for Pyrolytic-Carbon Coatings at 1500°F

Capsule No. and Coating	Ratio of R/B^a for Bare Particles to R/B for Coated Particles				
	Xe ¹³³	Xe ¹³⁵	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷
3M-SP-2 Laminar	140	50	37	28	20
HTM-1 Columnar	200	160	200	250	250
NCG-AD Duplex	2000	1100	1500	1250	1300

^aRelease rate/birth rate.

20.3 IRRADIATION EFFECTS ON METAL-CLAD FUELS

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ORR Prototype Capsules (Group IV)

Six stainless-steel-clad UO_2 , EGCR prototype, fuel elements were irradiated in the ORR poolside facility⁶ at temperatures of 1300 to 1600°F to burnups of 1000 to 3000 Mwd per metric ton of uranium. Capsules O1C and O8B were approximately 7 in. long $\times$ $\frac{3}{4}$ in. OD and were fitted with six clad thermocouples and one central thermocouple. These capsules were intended to serve as proof tests for EGCR instrumented fuel assemblies, to evaluate central thermocouple weld materials, and to study the effect of initial grain size of the UO_2 fuel pellets on fission-gas release. Of the four remaining capsules in group IV, O3, O4, and O7 were fabricated by swaging at 800°C, and O2 was fabricated by hand-tamping UO_2 powder into a $\frac{1}{2}$ -in. type 304 stainless steel tube. The purpose of these irradiations was to evaluate the irradiation behavior of bulk UO_2 under EGCR conditions in the form of both hot-swaged and tamp-packed fuel capsules.

The postirradiation examination of the first two capsules has previously been reported in detail.⁷ Examination revealed that O1C and O8B failed during irradiation. Cracks were found in the GE-81 braze joint in both capsules. Separation of the braze from the thermocouple sheath and the cracking of the braze material itself were evident. Severe void formation was found in the cladding adjacent to a missing piece of UO_2 pellet (see Fig. 20.24). Circumferential ridges at pellet-to-pellet interfaces were observed in the cladding. Voids seen in these ridges are believed to be the result of shear rather than hoop stresses. This was ascertained by determining metallographically the size and shape of the voids in both longitudinal and transverse directions of the clad, which showed the intergranular voids to be essentially of the same shape and size in both planes. If hoop stresses had been prevalent during clad deformation, the resultant voids would not have been alike in character in the two sections examined. Sigma phase was found in some areas of the cladding of both fuel elements. Since the sigma phase occurred only in a small, spherical precipitate, it was concluded that sigma phase does not cause the deleterious effect on the cladding during irradiation.

The swaged and tamp-packed fuel elements⁸ were irradiated to clad temperatures of 1300°F

⁵Metals and Ceramics Division.

⁶Irradiation experiments performed by the Reactor Division.

⁷D. F. Toner *et al.*, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, pp 67-76.

⁸D. F. Toner *et al.*, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, pp 76-83, and J. G. Morgan *et al.*, "Examination of Irradiated Capsules," *GCR Semiann. Progr. Rept. Sept. 30, 1962* (now in process).

Fig. 20.24. Longitudinal Section Through Depression in the Cladding of ORR Capsule OIC-4. Depression occurred at a pellet-to-pellet interface. Severe deformation resulted in intergranular voids in the type 304 stainless steel cladding. As polished, 75X. Reduced 33%.

and linear heat ratings of 8,000 and 15,000 Btu $\text{hr}^{-1} \text{ft}^{-1}$.⁹ Postirradiation examination revealed that none of the fuel elements failed during reactor operation. The tamp-packed capsule O2-4 contained UO_2 packed to 59% of theoretical density. The cladding showed no evidence of potential failure, even though a fine precipitate was observed throughout the cladding. Metallographic examination of the UO_2 showed that no sintering had occurred during operation. The stainless steel tube collapsed under the irradiation conditions, but this was in accordance with previous predictions.¹⁰

The examination of O3-4, swaged at 800°C and irradiated to a linear heat rating of 15,000 Btu $\text{hr}^{-1} \text{ft}^{-1}$, revealed that UO_2 had been forced into the cladding to a depth of approximately $\frac{1}{2}$ mil. Metallographic examination revealed evidence of sintering in the center of the fuel column, whereas there was no evidence of sintering at the outer surface.

⁹W. C. Thurber, "Compact Powder Fuel Irradiation Studies at Oak Ridge National Laboratory," presented at the Symposium on the Powder Packed UO_2 Fuel Element, Windsor, Connecticut, Nov. 30–Dec. 1, 1961.

¹⁰C. R. Kennedy and J. T. Venard, *Collapse of Tubes by External Pressure*, ORNL TM-166 (Apr. 17, 1962).

The examination of O6-4, swaged at 800°C and irradiated to a linear heat rating of 15,000 Btu $\text{hr}^{-1} \text{ft}^{-1}$, showed that a central void was present in the fuel column (see Fig. 20.25). Columnar grains began at the central void and extended past the midradius. A two-phase area, which has not yet been identified, was found at the outer surface of the UO_2 adjacent to the clad. The central void, present in this capsule and not in O3-4, is thought to have been caused by the poorer thermal conductivity and resultant higher fuel temperature due to a lower density UO_2 and a higher O to U ratio.

The postirradiation examination of O7-4, swaged at 800°C and irradiated to a linear heat rating of 8000 Btu $\text{hr}^{-1} \text{ft}^{-1}$, showed that a considerable amount of sigma-phase material was present at the inner surfaces of the cladding to approximately half-way across the wall. A shell surrounded the particles of UO_2 adjacent to the outer surface. This shell is thought to have been caused by excess oxygen present during the swaging of the fuel element. No evidence of sintering was observed.

Two important conclusions which may be drawn from this examination are that the sigma phase which has been observed in capsule claddings is not significant with respect to their irradiation

Fig. 20.25. Cross Section of Capsule 06-4, Showing a Central Void in the UO_2 .

integrity, and that the method of hot swaging and tamp-packing of UO_2 has proven to be acceptable for fuel elements irradiated to a burnup of 2500 Mwd per metric ton of uranium at a clad temperature of 1300°F .

Loop 1 Capsules. — Test elements 19 in. long and 0.75 in. in diameter containing hollow UO_2 fuel pellets clad in type 304 stainless steel were fabricated and were irradiated in the GCR-ORR loop No. 1 for periods of from 7 to 14 weeks at clad temperatures between 1300 and 1500°F .¹¹ This facility is operated by the Reactor Division. Elements 5, 6, 7A, and 7B were irradiated for 7 weeks and element 7C was irradiated for 14 weeks

at a linear heat rating of between 24,000 and 28,000 $\text{Btu hr}^{-1} \text{ft}^{-1}$, in a dynamic helium stream at 300 psi.

Postirradiation examination, which has been previously reported,¹² has shown that under the EGCR designed operating conditions the $\frac{3}{4}$ -in. hollow-pellet stainless-steel-clad fuel elements are satisfactory. In addition, it has been demonstrated that the cored pellets do not disintegrate even though cracked during irradiation, that the fuel element maintains dimensional stability, and that no reactions occur during irradiation between the UO_2 and the stainless cladding. However, a peculiar corrosion effect on the type 304 stainless steel shroud, clad, and lead tube has been noted.

The lead tube, which is immediately above the fuel element, and the shroud, which surrounds the fuel element, on experiment No. 5 were rusty and very brittle. Subsequent metallographic examination of these accessory parts of the experiment showed an intergranular fracture which penetrated the wall of both parts. A typical microstructure is shown in Fig. 20.26. At higher magnification it was seen that a corrosion product, probably an oxide (see Fig. 20.27), was present in the grain boundaries. Analysis of the rust showed that Fe, Cr, and Ni were the principal constituents and that smaller quantities of Bi, Zn, Au, Ag, Mn, Mo, Cu, and Si were present. Structurally the rust was the gamma $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$. Similarly, rust was observed on the insert of element 7A and on the shroud of element 7C.

Analysis of the temperatures and of the helium purities under which these experiments were operated indicated that the stainless steel which was attacked was between 1000 and 1200°F and that up to 800 ppm of hydrogen and 500 ppm of H_2O were present in the helium coolant stream. After the fuel elements had been removed from the reactor, they were encapsulated in a loading tube under 20 psig of nitrogen and stored in the ORR pool. On at least two occasions water was observed in the loading tube when the fuel element was brought into the hot cell. It is suggested that during reactor operation a slight sensitization of the stainless steel occurred in the temperature range from 1000 to 1200°F . Subsequently, when

¹¹J. K. Franzreb, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, p 66; D. F. Toner *et al.*, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, p 67; J. G. Morgan *et al.*, "Examination of Irradiated Capsules," *GCR Semiann. Progr. Rept. Sept. 30, 1962* (now in process).

¹²D. F. Toner *et al.*, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, pp 83-96.

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Fig. 20.26. Transverse Sample Taken from Near One of the Breaks in the Type 304 Stainless Steel Lead Tube from Element 5. Friability of the tubing was demonstrated when whole grains loosened during mounting and fell out during polishing. As polished. 100X.

the fuel element was put into the loading tube, water leaked in through the valve and came in contact with the fuel element. Gamma activity from the fuel element could cause dissociation of nitrogen and water to form nitric acid, and the nitric acid could then attack the sensitized areas of the shroud, lead tube, and fuel element that were in the temperature range indicated above. Stobbs and Swallow¹³ observed that a pressurized-water loop with an air-cooled annulus surrounding it was attacked by a concentrated nitric acid in the Materials Testing Reactor.

Further investigation of this corrosion problem is in progress.

ETR Prototype Capsules

Three ETR-irradiated capsules¹⁴ were examined. Fabrication and irradiation data are summarized in Table 20.5. The results of one experiment (E-12) were similar to those reported previously for ORR-irradiated capsules.¹⁵ There were no major changes in the appearance or structure of this capsule. However, the other two capsules were found to have suffered gross fractures in the stainless steel cladding, shown in Figs. 20.28 and 20.29. Dimensional measurements showed

¹³Stobbs and Swallow, *Met. Rev.* 7, 25 (1962).

¹⁴The capsules were fabricated by the Metals and Ceramics Division. The experimental assemblies were built and the operation was supervised by the Reactor Division.

¹⁵J. G. Morgan *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, pp 105-106.

Fig. 20.27. Corrosion Product Found in Fractures in Type 304 Stainless Steel Lead Tubes of Element 5. As polished. 1000X.

diameter increases up to about 0.060 in., or about 8% (capsule E-4). As a result of the fractures, no fission-gas release data were obtained, and NaK from the surrounding heat transfer baths had completely filled both capsules. When the capsules were disassembled, longitudinal voids were found in the centers of the originally solid fuel columns, and extensive columnar grain growth around these voids was apparent (Figs. 20.30 and 20.31). These phenomena are typical of sintered UO_2 which has been heated to very high temperatures in a steep thermal gradient.^{16,17}

Metallographic examination of fuel and cladding samples from capsule E-1A has been completed.

¹⁶V. B. Lawson and J. R. MacEwan, *Thermal Simulation Experiments with a UO_2 Fuel Rod Assembly*, CRFD-915 (March 1960).

¹⁷J. L. Bates, *Nucleonics* 19, 6 (June 1961).

There was no evidence that any portion of the UO_2 had been molten during irradiation. Since the region of columnar grain growth extended from the central void to about 0.15 in. from the outside surface of the fuel pellet, it is obvious that the maximum design temperature of 3700°F was exceeded. The UO_2 structure indicates that the maximum central temperature was $\sim 5000^\circ\text{F}$. In addition, the results of burnup analysis (Table 20.5) indicate that the capsule was overpowered more than 50%. The measured burnup was 5050 Mwd per metric ton of uranium compared with a design value of 3350 Mwd per metric ton of uranium. There was a slight reduction in the thickness of the cladding at the fractures. As shown in Fig. 20.32, a heavy precipitate was found along the inner surface and along the intergranular fractures. This precipitate, which extended from the inner surface of the cladding to about midwall throughout the capsule, appeared to be nitrides of

Table 20.5. Data Summary for ETR-Irradiated Prototype Capsules

Capsule No.	Type of Fuel Pellet	U^{235} Enrichment (%)	Design Heat Flux ($\text{Btu hr}^{-1} \text{ft}^{-2}$)	Average Clad Temperature ($^{\circ}\text{F}$)	Uranium Burnup ^a (Mwd/metric ton)
E-1A	Solid	0.71	159,000	1290	5050
E-4	Solid	0.71	159,000	1320	8300
E-12	Hollow-BeO bushing	1.0	153,000	1500	1140

^aBased on Ce^{144}/U ratio.

stainless steel. The probable source of the nitrogen was an abundance of UN_2 in the outer regions of the UO_2 fuel pellets.

Thus it is concluded that the primary causes of cladding failure in capsule E-1A were (1) formation of nitrides in the inner surface region of the cladding and (2) overheating of the UO_2 fuel. The presence of the nitrides tended to weaken and embrittle the cladding, making it more susceptible

to fracture when subjected to the unusual stress from the overheated fuel. The examination of capsule E-4 is incomplete.

Examination of Beryllium-Clad UO_2 Capsules

The examination of eight beryllium-clad UO_2 -fueled capsules, which was partially reported previously,^{18,19} has been completed. The United Kingdom AEA and the French CEA cooperated with ORNL in the preparation of these experiments. The work at ORNL was sponsored by the Gas-Cooled Reactor Program.

The capsules were irradiated in the ORR at a nominal clad temperature of 1100°F to uranium

Fig. 20.28. Overall View of Capsule E-1A, Showing Clad Fracture. 0.45X. Reduced 19%.

¹⁸J. G. Morgan *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, pp 106-10.

¹⁹M. F. Osborne *et al.*, *Trans. Am. Nucl. Soc.* 5, 43 (1962).

Fig. 20.29. Closeup View of Clad Fracture, Capsule E-4. Note pieces of sheathed thermocouple and foil thermocouple strap adhered to clad. 2.5X. Reduced 34%.

Fig. 20.30. Transverse Section of Capsule E-1A, Showing Clad Fracture and Central Void in UO_2 . 4.3X. Reduced 7%.

burnups of from 1000 to 4000 Mwd per metric ton, or about 0.1 to 0.4 at. %, of uranium. The results of the last two experiments were similar to those of the first six. Clad fractures during operation occurred only in the two capsules which were known to have substandard welds. Two other capsules, which had been subjected to the highest external pressure during irradiation (up to 725 psi), suffered stress-relief fracturing during disassembly in the hot cell.

No significant changes in the microstructure of the UO_2 were found during metallographic examination. However, there were deleterious effects on the beryllium cladding. There was general subsurface void formation in all interior regions which were in contact with the UO_2 . These voids had penetrated to a maximum depth of about 20 mils, more than half the wall thickness. No voids were found in the cladding adjacent to MgO end spacers. There was a reaction area at the interface between

the UO_2 pellet and the beryllium end plug in one of the capsules which did not contain MgO end spacers. This reaction had consumed about 15 mils of the beryllium end plug and had penetrated the grain boundaries in the UO_2 . The reaction product appeared to be UBe_{13} . Other investigators have not found such an extensive reaction at this temperature in out-of-pile tests.²⁰ On the basis of these relatively short-term experiments, beryllium does not appear to be a promising material for a high-temperature UO_2 -containing reactor fuel element.

Irradiation Effects on Fueled BeO

The dimensional stability of $\text{BeO-UO}_2\text{-ThO}_2$ was found to be somewhat better than that of

²⁰ A. G. Knapton and K.B.C. West, *J. Nucl. Mater.* 3(2), 239 (1961).

Fig. 20.31. Transverse Section of Capsule E-4, Showing Clad Fracture and Central Void and Circumferential Crack in UO_2 . 2.8X. Reduced 7%.

Fig. 20.32. Fracture in Cladding of Capsule E-1A. Note heavy precipitate along inner surface and fracture region. Etched. 250X.

BeO-UO₂ for irradiations to about 4 at. % of uranium plus thorium burnup at about 2000°F. The dimensional increase for the former was about 0.5%; for the latter the average increase was about 2%, with a maximum of 3.5%. With the exception of those capsules which leaked during irradiation, all fuel pellets were removed intact and none were broken during handling in the hot cell. Fission-gas release was low, 0.1 to 0.3% of the Kr⁸⁵ formed. There was no evidence of any chemical reactions in capsules which had inert atmospheres. There appeared to be considerable fission-fragment damage to the microstructure of the BeO matrix material in both types of fuel. Apparently, the finely dispersed fuel particles tended to maximize this damage and possibly to maximize physical swelling of the fuel pellets. These results indicate that dispersions containing larger fuel particles (~100 μ) might be more stable

dimensionally. With the exception of dimensional stability, the radiation stability properties of these dispersion fuels are equal to or superior to those of bulk UO₂.

These experiments have been reported in detail previously.²¹

20.4 CONTINUOUS RELEASE OF FISSION GAS DURING IRRADIATION OF UO₂

R. M. Carroll P. E. Reagan
T. W. Fulton

An in-pile facility is in operation for the study of the factors controlling the fission-gas release from

²¹J. G. Morgan and M. F. Osborne, *Radiation Effects on Fueled BeO*, ORNL TM-311 (Oct. 4, 1962).

UO₂ during irradiation.²² The fuel specimen is heated by its own fission power. The power output is regulated by moving the fuel into or out of the reactor neutron flux. At constant fission power the temperature of the specimen is controlled by air cooling. Fission gas from the UO₂ fuel is entrained in an inert sweep gas (normally helium) and carried outside the reactor for analysis. The neutron flux for any power level is found by using argon as the sweep gas and measuring the argon activation.²³ To prevent oxidation of UO₂ specimens from trace amounts of oxygen in the purified sweep gas, 3% hydrogen is added to the argon and helium.

Thin-Plate Fused-Crystal UO₂

Two thin plates of fused-crystal UO₂ were sandwiched together with thermocouples between them and with thermocouples also pressed against the outer surface. These specimens, obtained by slicing a lump of fused UO₂, had very large grains. The fission-gas release from the fused-crystal specimen was similar to that from a sintered specimen under the same conditions. This demonstrated either that the surface area of the high-density sintered specimen was almost equal to the geometric surface area of the polished fused

specimen or that total surface area is not the dominating factor for fission-gas release.

The steady-state fission-gas release from this specimen was measured over a series of different temperature ranges. These are summarized in Table 20.6. The last two measurements, after about nine weeks of irradiation, showed a definite increase of release rate. When the capsule was removed from the reactor and examined to determine the cause of this increase, it was found that the specimens were broken, apparently along the grain boundaries. The increased fission-gas release was probably caused by the increased surface area of the specimen.

Iodine release measurements are obtained by withdrawing the specimen from the reactor flux after an equilibrium release rate has been obtained. The continued production of Xe¹³³ and Xe¹³⁵ was from the decay of iodine that emerged from the UO₂ during irradiation and deposited within the sweep system. At steady-state operation, the escape of iodine from the UO₂ is equal to the production of xenon from the deposited iodine. The steady-state production of xenon can be determined (see Fig. 20.33) by extrapolating the measured release rate to the moment the sample is withdrawn.

Iodine was found to diffuse from the fused-crystal specimen with the same temperature dependence as xenon and krypton. This identical temperature dependency was also found for all previous specimens, regardless of composition. For the gas that was released, 48% of the 133 isobar and 75% of the 135 isobar were released as iodine. This same ratio of xenon-to-iodine release was found for a

²²R. M. Carroll and C. D. Baumann, *Experiment on Continuous Release of Fission Gas During Irradiation*, ORNL-3050 (Feb. 9, 1961).

²³R. M. Carroll, "Argon Activation Measures Irradiation Flux Continuously," *Nucleonics* 20(2) (1962).

Table 20.6. Fractional Fission-Gas Release from Fused-Crystal UO₂ Specimen
[(Release Rate/Production Rate) × 10⁴]

Temperature (°C)	Xe ¹³³	Xe ¹³⁵	I ¹³³	I ¹³⁵	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷
850	13	2.9	7.3	8.5	17	6.5	
912	16	3.9	14	16	23	7.8	
765	9.1	1.2	10	7	14	7.4	6.1
985	40	8.4	43	33	40	19	18
812	22	2.3	12	10	22	13	8.1
955	47	9.7	59	54	70	33	26

Fig. 20.33. In-Pile Fission-Gas Release from Fused Crystal of UO_2 During Reactor Scram; CI-7 Experiment.

sintered nonstoichiometric specimen,²⁴ although the absolute release rate was much greater for the sintered specimen. Apparently, anything affecting the release of xenon affects the iodine release in the same manner.

The fused-crystal specimen had a low (0.3%) enrichment and was irradiated at a neutron flux of $3.3 \times 10^{13} \text{ nv}$ to produce the desired fission rate. This flux was a factor of 10 higher than for the nonstoichiometric sintered specimen. Because of the difference in flux, it was expected that the 2.7×10^6 barn cross section of Xe^{135} would cause the $\text{Xe}^{133}/\text{Xe}^{135}$ ratio to increase with increasing flux. The ratio, however, remained the same for both specimens.²⁵ The only explanation, thus far, for this phenomenon is that the iodine precursor of the xenon dominates the diffusion of the iodine-xenon isobar. Thus the iodine might diffuse to

the surface of the UO_2 , and then, after the iodine decays to xenon, the xenon is released close to the surface with very little time for decay.

The activation energy for the fused-crystal specimens was the same as for the previous specimens, 30 to 40 kcal/mole. This was somewhat unexpected because of the different composition of the fused-crystal specimen.

Thin-plate specimens of sintered UO_2 , containing excess oxygen, released bursts of fission gas when the specimens were heated or cooled.²⁴ The fission-gas release from the fused-crystal specimen was also observed during temperature fluctuations to see if similar bursts were produced. Heating bursts were always produced, but no cooling bursts were found (Fig. 20.33).

Thin-Plate Single-Crystal UO_2

Thin-slab specimens of high-quality single-crystal UO_2 have been obtained from Hanford. These were sandwiched together with thermocouples between them and pressed against the outer surfaces as described above. The thermocouples served the added function of electrical

²⁴R. M. Carroll, "Continuous Release of Fission Gas from UO_2 During Irradiation," *Symposium on Radiation Effects in Refractory Fuel Compounds*, ASTM Special Technical Publication No. 306.

²⁵F. R. McQuilkin, *Gas-Cooled Reactor Program Quart. Progr. Rept. Mar. 31, 1962*, ORNL-3302, p 62.

probes to measure the electrical conductivity of the UO_2 during irradiation²⁶ (see Fig. 20.34). The electrical resistivity of crystalline UO_2 was decreased by a factor of 200 as soon as irradiation began. This decrease was due to ionizing irradiation rather than fission-product impurities since, in the initial portion of irradiation, the resistivity could be increased by removing the specimen from the flux. After 24 hr of irradiation, the change of resistivity was irreversible.

The activation energy for electrical resistivity changed dramatically from 11.3 eV (preirradiation) to 1.6 eV during the first few hours of irradiation, then stabilizing at 0.4 eV for subsequent irradiation. The values of resistivity and activation energy are compared in Table 20.7.

From these data we conclude that irradiation lowers the electrical resistivity by providing carriers for the electrical current. The activation energy is decreased because of irradiation-induced

disorder in the UO_2 structure. This effect is more pronounced in the case of a highly ordered structure such as the single-crystal specimen.

Fission-gas release decreased slightly after the first two weeks of irradiation. This was probably caused by reduction of the oxygen-contaminated surface by the hydrogen in the sweep gas. The fractional fission-gas release in the 700°C range is compared in Table 20.8 for a sintered fine-grain specimen, a fused-crystal large-grain specimen, and the single-crystal specimen.

The activation energy for Kr^{88} release is 46 to 50 kcal/mole for the single-crystal specimen. This is higher than the 30- to 40-kcal/mole activation energy for all previous specimens. The temperature-dependent gas release for the single-crystal specimen begins at about 630°C. This is about 100°C higher than for the sintered and the fused specimens.

The single-crystal specimen emits heating bursts, thus demonstrating that the heating burst is not a grain-boundary phenomenon. No cooling bursts have been emitted by this specimen.

²⁶M. D. Karkhanavala and R. M. Carroll, *In-Pile Measurement of the Electrical Resistivity and Thermoelectric Power of Sintered UO_2* , ORNL-3093 (April 1961).

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Fig. 20.34. Schematic of Arrangement Used for Measurement of Electrical Conductivity and Thermoelectric Power.

Table 20.7. Comparison of Resistivity and Activation-Energy Values

Specimen	Resistivity at 700°C (ohm-cm)		Activation Energy (ev)	
	Before Irradiation	After Irradiation to 10^{18} nvt	Before Irradiation	After Irradiation to 10^{18} nvt
Sintered, fine grain	2.0	0.8	0.65	0.50
Fused crystal, very large grain	1.7		0.52	
Single crystal	~500	2.5	11.3	0.4

Table 20.8. Steady-State Fractional Fission-Gas Release in 700°C Range (R/B)^a

Specimen	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷	Xe ¹³⁵	Xe ¹³³
Fine grain, sintered; 730°C	8.3×10^{-4}	7.5×10^{-4}		5.2×10^{-4}	1.5×10^{-3}
Large grain, fused; 765°C	14×10^{-4}	7.4×10^{-4}	6.1×10^{-4}	8.2×10^{-4}	1.9×10^{-3}
Single crystal; 712°C	1.3×10^{-4}	5.2×10^{-5}	3.7×10^{-5}	1.9×10^{-4}	3.1×10^{-4}

^aRelease rate/birth rate.

Thermocouple Comparisons: W-5% Re vs W-26% Re with Pt vs Pt-10% Rh. — A hydrogen-trace atmosphere must be used in these sweep experiments to avoid oxidizing the UO₂ specimen. A disadvantage in using hydrogen additions is that the hydrogen will react with the high-temperature junctions of the Pt vs Pt-10% Rh thermocouples, causing them to become brittle. Early failure of bare Pt vs Pt-10% Rh thermocouples is to be expected under these conditions. Nevertheless, the electrical resistivity tests described above require that bare-wire Pt vs Pt-Rh thermocouples be used.

For the single-crystal UO₂ specimens, three types of thermocouples are used: bare-wire Pt vs Pt-10% Rh, Pt vs Pt-10% Rh clad in Inconel, and bare-wire W-5% Re vs W-26% Re. For the pre-irradiation electrical conductivity test the assembled capsule (in a reducing atmosphere) was

heated in a furnace before being inserted into the reactor. At this time in the reducing atmosphere all thermocouples indicated the same temperature. During irradiation the W-5% Re vs W-26% Re thermocouples have indicated about 10% lower temperatures than the Pt vs Pt-10% Rh. This difference started immediately and has not changed appreciably. The amount of shift is temperature dependent, being least (about 6%) at 700 to 800°C and increasing at higher and lower temperatures. The specimen has not been irradiated at temperatures higher than 1000°C, and the platinum thermocouples, both bare and clad, indicated 13% higher values for this temperature than did the tungsten thermocouples.

In view of the identical temperatures from all thermocouples during bench testing in the hydrogen atmosphere, we must conclude that irradiation lowered the emf output of the tungsten thermocouples.

20.5 MARITIME REACTOR FUEL

J. G. Morgan

M. F. Osborne

Four additional maritime reactor fuel bundles, similar to those reported previously,²⁷ have been examined. Each of these fuel-rod bundles had been irradiated in the pressurized-water loop facility in the ORR.²⁸ Each bundle consisted of three fuel rods 18 in. long and 0.5 in. in diameter, as shown in Fig. 20.35. Each rod was made of type 304L stainless steel tubing containing fused and ground UO_2 .

The fuel rods were fabricated by two processes: (1) 0.75-in.-OD tubing containing UO_2 powder was swaged to the finished diameter and (2) 0.5-in.-OD tubes were filled with vibratorily compacted UO_2 . Uranium dioxide densities of about 90% of theoretical were attained by both methods.²⁹ Fuel enrichment ranged from 0.80 to 2.83% U^{235} .

The rods were cooled by high-purity water at 1750 psi and 500°F, flowing at about 10 fps. The experiments were irradiated to burnups of up to 4600 Mwd per metric ton of uranium, with center-fuel temperatures estimated to be as high as 5000°F and a maximum heat flux of 400,000 $\text{Btu hr}^{-1} \text{ft}^{-2}$.³⁰

There was no change in the exterior appearance of the irradiated rods except for the accumulation of a dark oxide film. Radiochemical analysis of this film showed that the principal components were stainless steel corrosion products: Cr, Fe, Mn, and Co. The bundles and the individual rods were measured for diameter changes and bowing. No significant rod-bowing changes were found. Diameter measurements indicated that one fuel rod was 0.007 to 0.010 in. smaller after irradiation, and the diameter change of another rod varied from +0.011 to -0.005 in. Diameter changes for the other ten rods were within experimental uncertainty.

Samples of the gas from each rod were analyzed for both stable and radioactive components. The fractions of the Kr^{85} released from the fuel, based on measured burnup, are shown in Table 20.9. As expected, the fission-gas release increased with irradiation temperature and burnup, reaching a maximum of 23%. Gamma scans of the fuel rods were similar to that reported previously;²⁷ no unusual distributions of radioactivity were found. The ratios of peak-to-average radioactivity varied from 1.14 to 1.23, indicating relatively uniform burnup along the length of the rod.

The fuel rods were sectioned transversely and longitudinally for examination of the fuel. The appearance of the fuel in a typical rod from experiment No. 3 is shown in Fig. 20.36. Transverse cracks in the fuel are correlated with irregularities in the gamma scan. One fuel rod, denoted 601, contained a void in the center of the fuel column (Fig. 20.37). The void was about 6 in. long, and its maximum diameter was about 0.04 in. This type of central void, with the accompanying growth of columnar grains around it, is formed in UO_2 when steep thermal gradients exist at temperatures

²⁷J. G. Morgan, M. F. Osborne, and H. E. Robertson, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 112; J. G. Morgan and M. F. Osborne, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1961*, ORNL-3213, p 113.

²⁸J. A. Colins, J. K. Franzreb, and D. E. Tidwell, *Maritime Reactor Program Ann. Progr. Rept. Nov. 30, 1961*, ORNL-3238, p 41.

²⁹J. T. Lamartine, *Preirradiation Data: Maritime ORR Loop Experiments 4, 5, and 6*, ORNL CF-62-3-88 (Mar. 28, 1962).

³⁰V. O. Haynes, *ORR Pressurized-Water Loop Experiments: General Description and Operation Information for Experiments 4, 5, and 6*, ORNL CF-61-2-92.

Fig. 20.35. Schematic of Experimental Fuel-Rod Bundle, Maritime Reactor Program.

Table 20.9. Data Summary for Maritime Reactor Fuel Examinations

Experiment No.	Rod No.	Fabrication Method	Maximum Heat Flux ^a (Btu hr ⁻¹ ft ⁻²)	Maximum Fuel Temperature ^a (°F)	Maximum Burnup ^b (Mwd per metric ton of uranium)	Kr ⁸⁵ Release ^c (%)	Plutonium Concentration (mg of Pu per g of U)
3	3A1	Cold swaged	100,000	2200	2480	3.7	1.74
	3B1	Cold swaged	100,000	2700	3030	3.4	
	3C1	Cold swaged	100,000	2700	3320	7.6	
	3N1	Cold swaged	100,000	2200	2950	3.3	
	3O1	Hot swaged	120,000	2200	2900	3.1	
	3P1	Hot swaged	120,000	2700	3820	3.2	
4	4N1	Cold swaged	200,000	3800	3120	4.1	1.65
	4O1	Vibratorily compacted	275,000	2900	2870	2.2	1.87
	4P1	Vibratorily compacted	275,000	2900	2440	2.9	
6	6N1	Vibratorily compacted	400,000	4500	3750	4.7	1.34
	6O1	Vibratorily compacted	400,000	4500	4660	23.4	1.58
	6P1	Vibratorily compacted	400,000	5000	4150	10.5	

^aCalculated.^bBased on Ce¹⁴⁴/U ratio.^cBased on average burnup.

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Fig. 20.36. Correlation of Gamma Radioactivity Scan with Section of Maritime Fuel Rod. Note dips in gamma scan adjacent to fractures in UO₂ fuel; section of fuel rod was cut longitudinally to expose fuel.

Fig. 20.37. Transverse Section of Maritime Rod 601, Showing Central Void and Columnar Grain Growth. Original appearance of fuel was similar to that near clad.

greater than 3000°F.^{31,32} Partial sintering of the UO_2 was observed in several other fuel rods which experienced temperatures greater than 3000°F.

In order to evaluate the breeding efficiency of this fuel, some samples were analyzed by mass spectrometry for the plutonium content. These results, shown in Table 20.9, agreed fairly well with values calculated from the neutron flux.

20.6 FUEL CYCLE PROGRAM³³

J. G. Morgan

M. F. Osborne

Postirradiation Examination

The primary objective of the Fuel Cycle Program is the development of suitable techniques for remote reprocessing and fabrication of reactor fuel containing U^{233} .³⁴ Test fuel rods of vibratorily

compacted UO_2 - ThO_2 powder in type 304 stainless steel tubing (Fig. 20.38) have been irradiated, and examinations are in process. Fuel prepared by two different processes is being studied. Fused and ground 4.5 wt % UO_2 fuel was obtained from Spencer Chemical Company, and material of the same composition was fabricated at ORNL by the "sol-gel E" process, which produces the oxide by chemical derivation. All uranium was fully enriched, that is, about 93% U^{235} .

Preliminary postirradiation data have been obtained for three rods irradiated in the MTR. These data are summarized in Table 20.10. One rod, designated MTR-43-41, contained sintered fuel pellets for direct comparison with the powder fuels. There was no significant difference in rod appearance after irradiation, and dimensional changes were small. The maximum bowing change was equal to the maximum bow of the rod before irradiation. The maximum diameter change was an increase of about 0.003 in.

Gamma scans indicated a burnup gradient along the rods, as shown in Fig. 20.39. Samples of the fission gas were analyzed by mass and gamma-ray spectrometry. Fractional release values for Kr^{85} were low, as shown in Table 20.10.

³¹V. B. Lawson and J. R. MacEwan, *Thermal Simulation Experiments with a UO_2 Fuel Rod Assembly*, CRFD-915 (March 1960).

³²J. L. Bates, *Nucleonics* **19**, 6 (June 1961).

³³Administered by the Metals and Ceramics Division.

³⁴S. A. Rabin, *Status of the Fuel Cycle Irradiation Program at ORNL*, ORNL CF-62-7-55.

Each rod was sectioned for fuel examination. No change in the appearance of the vibratorily compacted fuel was seen. As expected, the sintered fuel pellets were severely cracked. Samples of the fuel were analyzed for uranium and thorium burnup by radiochemical and mass

spectrometric techniques. [Thorium is consumed by the process $\text{Th}^{232}(n,\gamma)\text{Th}^{233} \xrightarrow{\beta^-} \text{Pa}^{233} \xrightarrow{\beta^-} \text{U}^{233}(n,f).$] The fuel is being examined metallographically.

Table 20.10. Data Summary for Fuel Cycle Rods

Experiment No.	Type of Fuel	Uranium Content (wt %)	Fuel Density (% of Theoretical)	Heat Flux ($\text{Btu hr}^{-1} \text{ft}^{-2}$)	Burnup ^b (Mwd per metric ton of metal)	Kr ⁸⁵ Release (% of Total)
MTR-43-41	Solid pellet	3.88	92.5	625,000	33,000	c
MTR-43-59	Fused and ground	3.96	85.7	570,000	11,000	2
MTR-43-63	Sol-gel	4.0	86.2	570,000	13,000	<1

^aCalculated.

^bBased on mass spectrometer measurements.

^cGas sample lost.

21. Polymers

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Differences in the Effects of Gamma and Reactor Radiation on Polystyrene

The previous report¹ presented measurements demonstrating that, for equal energy deposition, the mixed gamma radiation and neutrons of the ORNL Graphite Reactor produced 2.4 times as many cross-links in polystyrene as did gamma radiation alone. Other workers have found that energetic particles and gamma radiation produce about the same effects in simple aliphatic hydrocarbons.^{2,3} Recent evidence indicates that in aromatic liquids, product yields differ for heavy particles (e.g., neutrons and alphas) and gamma radiation.^{4,5} Therefore it is desirable to investigate the differences in molecular structure and in the volatile radiolysis products from polystyrene resulting from irradiation in a gamma source and in a reactor. Radiation-induced changes in the molecular structure of polystyrene are indicated by infrared spectra before and after irradiation. The volatile radiolysis products are being analyzed by gas chromatographic methods.

Infrared Analysis. — For infrared measurements, polystyrene films about 0.001, 0.002, and 0.004 in. thick have been irradiated up to doses of 2.5×10^{23} ev/g in a water-cooled Co⁶⁰ gamma source and in

the ORNL Graphite Reactor. The spectra show that there is a loss of phenyl groups as indicated by the reduction in the C-H stretching bands of the aromatic ring at 3088 and 3065 cm^{-1} and by the reduction in aromatic C-H bending modes at 1030 and 905 cm^{-1} . The reduction in these bands after 2.5×10^{23} ev/g amounts to about 10% of the original optical density, but is not large enough to show conclusively differences between reactor irradiation and gamma irradiation.

New bands appear at 865, 820, and 675 cm^{-1} , and these peaks show a greater increase for reactor irradiation than for gamma. The 865 and 820 bands have not been identified with a definite molecular species, but the 675 band corresponds very closely to the 671- cm^{-1} band for out-of-plane C-H bending in benzene.⁶ This band also has about the same frequency as a similar bending mode (680 to 700 cm^{-1}) in cis olefin groups. The narrow width of the band, however, is much more similar to the sharp benzene band than to the broad band of the olefin group.

The reduction in phenyl groups could result from rupture of the aromatic ring to form olefinic side chains. Reduction of phenyl groups could also result from breakage of the bond between the main chain and the benzene ring, followed by the evolution of benzene. The latter possibility will be studied by observation of the 675- and 1030- cm^{-1} bands when irradiated samples are heated under vacuum. The latter band is assigned to corresponding C-H bending modes of both the benzene ring and the polystyrene aromatic ring.

A marked difference between gamma- and reactor-irradiated samples is the rate of oxidation

¹W. W. Parkinson *et al.*, *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, p 92.

²H. A. Dewhurst and R. H. Schuler, *J. Am. Chem. Soc.* **81**, 3210 (1959).

³A. E. DeVries and A. O. Allen, *J. Phys. Chem.* **63**, 879 (1959).

⁴W. G. Burns, W. Wild, and T. F. Williams, *Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1958* **29**, 266 (1958).

⁵T. Gaumann and R. H. Schuler, *J. Phys. Chem.* **65**, 703 (1961).

⁶N. Herzfeld *et al.* *J. Chem. Soc.* **1946**, 272.

upon exposure of the samples to air after irradiation. The growth of infrared bands at 3400 and 1710 cm^{-1} , characteristic of the -OH and >CO groups, respectively, is an indication of oxidation rate. These bands are negligible in samples which have received 2.5×10^{23} ev/g of gamma radiation. In contrast, samples receiving equivalent doses of mixed reactor radiation show absorptivities in these bands of 3 to 4 cm^{-1} after 45 days in air.

Gas Chromatographic Analysis. — Samples of polystyrene were irradiated up to doses of 1.8×10^{23} ev/g in a water-cooled Co^{60} source having a dose rate of 3×10^{20} $\text{ev g}^{-1} \text{hr}^{-1}$. The retention characteristics of the gas chromatographic columns made it necessary to separate the volatile products into condensable and noncondensable fractions before analysis. This was accomplished by transferring the products through a detachable U-shaped trap immersed in liquid nitrogen and then into a Toepler pump. After irradiation all samples were annealed for 4 hr at 100°C before being opened and were maintained at this temperature during transfer of radiolysis products to reduce the solubility of gaseous products in the polystyrene.

The noncondensable fraction was collected in the Toepler pump, where its volume was measured. Gas chromatographic analysis, using a Perkin-Elmer model 154 vapor fractometer with a thermal conductivity detector and a silica gel column, revealed that the noncondensable fraction consisted essentially of hydrogen with trace amounts of methane too small to measure. The Toepler pump therefore afforded a convenient means of measuring the amount of radiation-produced hydrogen in each sample.

The condensable fraction was introduced into the chromatograph and was found to consist entirely of benzene. The identification was carried out by comparing the retention times of benzene with those of the unknown from two separate runs: one on a column of didecyl phthalate on Celite and the other on a column of triphenyl phosphate on Celite. Measurement of the height of the benzene peak allowed a quantitative determination of benzene once the response of the chromatograph had been calibrated in terms of the amount of benzene required to produce a given peak height.

In order to calibrate the chromatograph for benzene, the system⁷ shown in Fig. 21.1 was assem-

bled for introducing small known amounts of benzene vapor into the chromatograph. The apparatus consisted of a reservoir of benzene maintained at a temperature of $20.00 \pm 0.05^\circ\text{C}$, a gas burette, a mercury manometer, and a detachable U-shaped trap. Benzene vapors were allowed to expand into a known volume in the gas burette, the vapor pressure was measured, and the vapor was condensed into the trap with liquid nitrogen. The trap was then removed from the system, attached to the chromatograph, and the contents were flushed onto the column with the carrier gas.

Considerable difficulty was encountered in choosing a suitable stopcock grease for use in contact with benzene vapor. Various fluorocarbon, silicone, polyethylene, and other hydrocarbon greases were tried and found unsatisfactory due to absorption of benzene vapor. A water-soluble grease (Phynal, made by Podbielniak, Inc., Chicago, Illinois) was found to be quite satisfactory, provided traces of water were removed from the grease prior to use. With the use of this grease and the system described, it was possible to introduce known amounts of benzene into the chromatograph with a reproducibility of 2% or better in the range of 0 to 15×10^{-6} mole of benzene.

The analytical results for several gamma-irradiated polystyrene samples are presented in Table 21.1. The values of $G(\text{H}_2)$ are in relatively good agreement with the value of 0.0223 found by Wall and Brown, who studied gas production from polystyrene and deuterated polystyrenes subjected to gamma radiation.⁸

With regard to benzene formation, Wall and Brown concluded that what benzene they observed arose from residual traces of benzene dissolved in the polymer prior to irradiation because their polystyrene samples were purified by precipitation from benzene solution. As a result of the present investigation, however, it appears that benzene production may indeed be radiation induced. The facts that benzene formation increases with dose and that blank runs, in which unirradiated samples were treated in exactly the same fashion as irradiated ones, yielded no detectable amounts of benzene tend to support this contention. Experiments involving samples irradiated to higher doses are in progress to clarify this point.

⁷P. S. Rudolph, private communication.

⁸L. A. Wall and D. W. Brown, *J. Phys. Chem.* **61**, 129 (1957).

Fig. 21.1. Apparatus for Transferring Known Amounts of Benzene Vapor.

A Versatile Gamma-Irradiation Facility. — In order to irradiate specimens of plastics and rubbers while mechanical properties are being measured over a broad range of temperatures, a source is required which has an intensity of at least 10^6 r/hr, uniform over a region of about 2 in. in diameter and 2 in. in height, and with maximum accessibility above and around the intense region. To meet these requirements a facility comprised of a cobalt source in a shielded compartment 7×10 ft and 7 ft high has been designed. The shielded compartment is designed with concrete walls adequate for 20,000 curies of cobalt.

The source itself is composed of cobalt pellets enclosed in tubes to form cylindrical source assemblies. These assemblies are arranged vertically around a circle of $2\frac{7}{16}$ in. in diameter to give a cylindrical source array. The source array is mounted on a screw jack by which the source can be retracted into a hole in the floor of the shielded compartment. A lead shielding plug is moved horizontally to close the storage hole

when the source is withdrawn, thus permitting the positioning of specimens and apparatus in the compartment for irradiation.

Table 21.1. Gas Production
from Gamma-Irradiated Polystyrene

Dose (ev/g)	H ₂ (mole/g)	C ₆ H ₆ (mole/g)	G(H ₂)	G(C ₆ H ₆)
$\times 10^{22}$	$\times 10^{-6}$	$\times 10^{-7}$	$\times 10^{-2}$	$\times 10^{-4}$
2.16	6.36	1.65	1.77	4.60
2.16	6.23	1.44	1.74	4.01
2.15	6.38	1.31	1.79	3.67
4.86	12.6	5.29	1.56	6.54
4.97	12.7	5.73	1.54	6.94
8.47	23.1	10.0	1.64	7.11
9.91	24.6	9.58	1.49	5.82
18.1	37.3	12.3	1.24	4.09

22. Ceramics

C. D. Bopp O. Sisman
R. L. Towns

Thermal Analysis Apparatus

Equipment has been developed which is suitable for thermal analysis of small samples (0.01 to 10 g) of a poor thermal conductor. The method is similar to that of Allison;¹ however, modifications have been introduced with respect to the places of measurement of temperature and to the method of calibration. A portion of the heat shield (Fig. 22.1) is removable to facilitate the loading of the crucible. After a sample is loaded, the heat shield is placed in a furnace, which is heated at a uniform rate, and curves are taken of both the temperature difference between the crucible and the heat shield, δ , vs time, t , and the temperature of the crucible vs time.

A convenient method of calibration² employs Peltier power which is obtained by passing a given value of direct current for a given time through a thermocouple junction attached to the crucible. The area under the resulting peak in the δ vs t curve is determined; the current is reversed, and the procedure repeated. The difference between the two areas equals twice the area equivalent to the Peltier power. In this method of calculation, the Joule power is eliminated by cancellation. If the Peltier current is applied for a time sufficiently long to reach a steady state, the labor of computing the area under the peaks is avoided. To a good approximation the area is proportional to energy if δ does not exceed a few degrees.

The Peltier method of calibration was tested by determining the heat of fusion of NaCl. A 50-mg

sample was sealed in a silica glass capillary and loaded into the crucible. The peak in the δ vs t curve was recorded when the furnace was heated uniformly past the fusion temperature (804.3°C) at a uniform rate of about 2°C per minute. The furnace temperature was then held steady at the average temperature of the fusion peak, and the apparatus was calibrated by the Peltier method. The area under the fusion peak, within the reproducibility of repeated trials (about 5%), was equivalent to the accepted value of the heat of fusion (124 cal/g).

If it is desired to obtain the specific heat or the heat of a reaction which does not give a sharp peak, a more extensive procedure is required. At the same rate of heating, δ vs t curves are obtained for the crucible plus the unknown and for the empty crucible. From a plot of the Peltier calibration vs temperature, the difference between the two curves may be interpreted in terms of the apparent specific heat of the unknown.

Nickel proved superior to copper or Monel as the material of construction of the heat shield and crucible, because oxidation of the metal surfaces and the formation of metallic deposits on the insulators were minimized. A vacuum furnace is used to reduce oxidation of the apparatus. At 25°C a pressure of less than about 0.1 μ was required to eliminate pressure dependence of the calibration (from heat transfer by convection). The calibration remained constant within the reproducibility of about 5% except on heating above about 900°C. By heating to above this temperature and then cooling to a lower temperature, the calibration (in terms of energy per unit area) was increased, but returned to the former value on standing at low temperature.

¹E. B. Allison and (Mrs.) J. Taylor, *Trans. Brit. Ceram. Soc.* 54, 677 (1955).

²W. Steiner and O. Krisement, *Arch. Eisenhuettenw.* 32, 701 (1961).

Fig. 22.1. Thermal Analysis Apparatus.

Radiation Stability of a Zircon Ceramic

Irradiation-induced changes in density, thermal conductivity, and Young's modulus for a zircon ceramic were previously described.³ Thermal analysis and x-ray diffraction⁴ were employed in a

further study. Reactor irradiation causes the material to resemble metamict zircon⁵⁻⁸ in the effects produced by heating. Heating produces partial annealing at relatively low temperature, decomposition at higher temperature, and re-formation of zircon at still higher temperature. According to the degree of irradiation, not all of these processes may occur.

For the range of radiation exposure studied (Table 22.1), the x-ray diffraction patterns of all but the sample with the lowest exposure were completely amorphous before heating. The change in the hydrostatically measured density is used to rank the exposures listed in Table 22.1, since this property is easy to measure and the accuracy is probably better than that for the flux estimates. The two highest exposures, for which the density change was saturated, are designated by listing the density change followed by "a" or "b." The density change correlates well with the thermal analysis data, as will be seen. The flux estimates are more accurate for the irradiation facility in the graphite reactor, since the variation with position and the variation during irradiation (from burnup of

³O. Sisman, C. D. Bopp, and R. L. Towns, *Solid State Div. Ann. Progr. Rept. Aug. 31, 1957*, ORNL-2413, p 80.

⁴Debye-Scherrer method by M. C. Wittels and F. A. Sherrill.

⁵M. V. Akhmanova and L. L. Leonova, *Geokhimiya* 1961, 401.

⁶H. D. Holland and D. Gottfried, *Acta Cryst.* 8, 291 (1955).

⁷M. V. Stackelberg and K. Chudoba, *Z. Krist.* 97, 252 (1937).

⁸J. Orcel, *Compt. Rend.* 236, 1052 (1953).

Table 22.1. Irradiation of Zircon

Density Decrease (%)	Reactor Type	Integrated Neutron Flux (neutrons/cm ²)	
		Thermal	Epithermal
		$\times 10^{20}$	$\times 10^{20}$
6.7	Light water	5	0.5
7.8	Graphite	1.3	1.0
10a	Graphite	2.0	1.6
10b	Light water	30	3

fuel) are less. The temperature of irradiation was less than 100°C.

The kinetic energy of fission fragments from thermal-neutron-induced fission of trace elements (uranium, 0.022%; thorium, 0.035%)⁹ is severalfold greater than energy from fast-neutron scattering, but, since the relative efficiency of the two types of radiation is unknown, it is uncertain whether or not fission fragments cause the major portion of the radiation change. It appears from Table 22.1 that the density change may be corre-

lated with the epithermal flux. However, the epithermal fluxes in the two irradiation facilities are not directly comparable, since the average energy is higher in the facility in the light-water reactor.

Three exothermic peaks were found in the apparent specific heat of the irradiated zircon (Table 22.2). All three were absent when a sample was

⁹Uranium by delayed-neutron activation analysis, F. F. Dyer; thorium by activation analysis, W. T. Mullins.

Table 22.2. Thermal Analysis of Irradiated Zircon

Threshold Temperature of Peak (°C)	Irradiation-Induced Density Decrease (%)	Energy Under Peak (cal/g)
200 ± 30	6.7	0.5 ± 0.3
200 ± 30	10a	0.03
(Low-temperature peak not detected)	10b	
880 ± 10	6.7	15
880 ± 10	7.8	15
880 ± 10	10a	16
880 ± 10	10b	16
1090	6.7	25 ± 10
(High-temperature peak not detected below 1200°C)	7.8	
(High-temperature peak not detected below 1200°C)	10a	

Table 22.3. X-ray Diffraction Patterns of Irradiated Zircon^a

Heat Treatment	Irradiation-Induced Density Decrease (%)	Description
None	6.7	Extremely broad lines
None	7.8	Amorphous
After heating to 400°C	6.7	Moderately broad zircon lines; no zirconia lines
After heating to 900°C	10b	Moderately broad zirconia lines; no zircon or quartz lines
After heating to 1100°C	6.7	Sharp zircon lines; no zirconia lines

^aSee ref. 4.

heated a second time. The threshold temperatures of the peaks are 200, 880, and 1090°C. The decrease in line breadth (Table 22.3) associated the 200°C peak with annealing of a part of the radiation change. This peak was absent in the most highly irradiated sample. The 880°C peak is identified with the decomposition of zircon to zirconia (however, both the zircon and zirconia patterns show broadened lines). Decomposition

was undergone by all the samples studied, and there was small effect of the degree of irradiation on the temperature or energy of the reaction. The 1090°C peak, which is identified with the formation of zircon from zirconia and silica, was found only for the lowest radiation exposure. The absence of this peak in the more highly irradiated samples is probably related to the lower density of such samples (Table 22.4).

Table 22.4. Density (at 25°C) After Decomposition

Irradiation-Induced Density Decrease (%)	Density Before Heating (g/cm ³)	Density After Heating to 900°C (g/cm ³)
Unirradiated	3.70	3.76
7.8	3.44	3.62
10a	3.36	3.47
10b	3.38	3.39

23. Radiation Dosimetry

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W. K. Kirkland

O. Sisman
W. W. Parkinson

The use of mixtures of a hydrocarbon and halogenated compounds to measure radiation dose by means of hydrogen ion production was described earlier.¹ By selection of mixtures having the minimum and maximum hydrogen content, it should be possible to prepare dosimeters sensitive only to gamma radiation in one case and sensitive to both gammas and fast neutrons in the other case.

A solution of low hydrogen content consisting of 0.3 wt % $C_{10}H_{22}$ and 0.3 wt % $C_2H_2Cl_4$ in C_8F_{16} would receive no more than 5% of its total dose from interaction with neutrons when exposed to the flux of most reactors. This mixture gave hydrogen ion yields of poor reproducibility in a series of irradiations in a gamma source. The variation in yields appears to have resulted from variations in moisture condensed in the solution by the chilling process employed during the evacuation and sealing of the containers. Efforts to reduce water contamination did not improve the reproducibility of yields, but the introduction of a slight excess of water over saturation resulted in consistent yields. Since the solubility of water in the mixture is low, the added water does not increase the interaction with fast neutrons appreciably. The acid produced in wet dosimeter mixtures having about 0.002 g of water per gram of solution is plotted in Fig. 23.1.

For a dosimeter solution sensitive to both fast neutrons and gamma radiation, solutions of 0.3 wt % $C_2H_2Cl_4$ in decane ($C_{10}H_{22}$) were prepared. Specimens were irradiated in gamma sources at room temperature and at 80°C and at two intensities to measure the linearity and the dependence on temperature and intensity. The hydrogen ion or acid yields are shown in Fig. 23.2. The high

Fig. 23.1. Acid Production in Wet C_8F_{16} - $C_{10}H_{22}$ - $C_2H_2Cl_4$.

Fig. 23.2. Acid Production in $C_{10}H_{22}$ - $C_2H_2Cl_4$.

¹C. D. Bopp et al., *Solid State Div. Ann. Progr. Rept.* Aug. 31, 1961, ORNL-3213, p 101.

yields at 80°C indicate that in the reaction mechanisms of acid production, there is at least one rate-controlling process which is temperature dependent, perhaps a diffusion process or atomic abstraction step. The lower yield at high intensity suggests that in the overall reaction there is a step involving the disappearance of an active species, without producing hydrogen ions, which is bimolecular and, hence, intensity dependent.

These intensity- and temperature-dependent steps are probably inherent in the acid production re-

actions in a hydrocarbon solvent. The possibility of altering the acid production reactions was investigated by adding water and ethanol to the dosimeter solution. Ethanol was added to make up 21.1 wt % to permit dissolving 0.21 wt % water. Irradiations in progress indicate that dependence on temperature may be reduced, but not eliminated. Figure 23.3 presents the acid production as a function of dose for dosimeter solutions composed of 78.4 wt % $C_{10}H_{22}$, 21.1 wt % C_2H_5OH , 0.24 wt % $C_2H_2Cl_4$, and 0.21 wt % H_2O .

Fig. 23.3. Acid Production in $C_{10}H_{22}$ - C_2H_5OH - $C_2H_2Cl_4$ - H_2O .

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Publications and Papers

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Publications and Papers

- R. F. Bass, J. W. Cleland, and J. H. Crawford, Jr.
 "Radiation Effects on Deep-Level Impurity States in Ge," *Bull. Am. Phys. Soc.* **6**, 461 (1961).
- C. Bassani, M. S. Wechsler, J. M. Williams, J. H. Barrett, and R. H. Kernohan
 "The Dependence of Radiation-Enhanced Diffusion in Cu-Al on Neutron Flux and Energy Spectrum," *Bull. Am. Phys. Soc.* **7**, 170 (1962).
- R. G. Berggren
 "Critical Factors in the Interpretation of Radiation Effects on the Properties of Metals," presented at 1962 Nuclear Congress, New York, June 4-7, 1962.
- D. S. Billington and D. O. Thompson
 "Radiation Damage Research in Low-Power Nuclear Reactors," presented at International Atomic Energy Agency Symposium on the Programming and Utilization of Research Reactors, Vienna, Austria, Oct. 16-20, 1961.
- T. H. Blewitt and R. R. Coltman
 "Cryogenic Techniques for Irradiation Studies in Nuclear Reactors," p 274 in *Experimental Cryophysics* (ed. by F. E. Hoare *et al.*), Butterworths, London, 1961.
- T. H. Blewitt, R. R. Coltman, C. E. Klabunde, and D. L. McDonald
 "Low Temperature Resistivity of Sodium and the Effects of Nuclear Irradiation," *J. Appl. Phys.* **33**, 1612 (1962).
- C. D. Bopp and R. L. Towns
 "Calorimetric Measurement of Nuclear Heating in a Reactor," *Nucl. Sci. Eng.* **13**, 245 (1962).
- R. H. Busey and E. Sonder
 "Magnetic Susceptibility of Potassium Hexachlororhenate (IV) and Potassium Hexabromorhenate (IV) from 5 to 300°K," *J. Chem. Phys.* **36**, 93 (1962).
- R. M. Carroll
 "Argon Activation Measures Irradiation Flux Continuously," *Nucleonics* **20**(2), 42 (1962).
 "Continuous Release of Fission Gas from UO_2 During Irradiation," p 110 in *Symposium on Radiation Effects in Refractory Fuel Compounds*, ASTM-STP-306, 1961.
- J. G. Castle, Jr., D. W. Feldman, and R. A. Weeks
 "Spin-Lattice Relaxation of E'_1 Centers in SiO_2 ," *Bull. Am. Phys. Soc.* **7**, 188 (1962).
- J. W. Cleland, R. F. Bass, and J. H. Crawford, Jr.
 "The Nature and Yield of Radiation-Induced Defects in Semiconductors," *Bull. Am. Phys. Soc.* **7**, 186 (1962).
- R. R. Coltman
 "Reactor Irradiation Studies at 4°K," presented at International Atomic Energy Agency Symposium on Radiation Damage in Solids and Reactor Materials, Venice, Italy, May 7-11, 1962.
 "Thermal and Fast Neutron Damage in Metals," presented at Southeastern Section Meeting, American Physical Society, Tallahassee, Florida, Apr. 5-7, 1962.

- T. F. Connolly
Operation of Research Materials Information Center, ORNL-TM-295 (July 30, 1962).
- J. H. Crawford, Jr.
 "Radiation Stability of Nonmetallic Structures," *Progr. Nucl. Energy, Ser. V*, **3**, 371 (1961).
- J. H. Crawford, Jr., and J. W. Cleland
 "Transmutation Doping and Recoil Effects in Semiconductors Exposed to Thermal Neutrons," p 269 in *Radioisotopes in the Physical Sciences and Industry*, International Atomic Energy Agency, Vienna, 1962.
- O. L. Curtis, Jr.
 "Effect of a Phase Transformation on the Vapor Phase Growth of Single-Crystal HgS ," *J. Appl. Phys.* **33**, 2461 (1962).
- O. L. Curtis, Jr., and J. H. Crawford, Jr.
 "Carrier Recombination and Trapping Processes in Irradiated Germanium," *Discussions Faraday Soc.* **31**, 107 (1961).
 "Radiation-Induced Recombination and Trapping Centers in Germanium. I. The Nature of the Recombination Process," *Phys. Rev.* **124**, 1731 (1961).
 "Radiation-Induced Recombination and Trapping Centers in Germanium. II. Annealing in Gamma-Irradiated, Antimony- and Arsenic-Doped Material," *Phys. Rev.* **126**, 1342 (1962).
- R. Gwin
 "Studies of the Scintillation Process in CsI(Tl) ," thesis, submitted to the Graduate Council of the University of Tennessee in partial fulfillment of the requirements for the Ph.D. degree, 1962.
- R. Gwin and R. B. Murray
 "Studies of the Scintillation Process in CsI(Tl) ," *IRE Trans. Nucl. Sci.* **NS-9**, 28 (1962).
- D. K. Holmes
 "Approximate Methods for Treating Annealing Processes Which Involve a Spectrum of Activation Energies," p 449 in *Chemical Effects of Nuclear Transformations*, vol I, International Atomic Energy Agency, Vienna, 1961.
 "The Ranges of Energetic Atoms in Solids," presented at International Atomic Energy Agency Symposium on Radiation Damage in Solids and Reactor Materials, Venice, Italy, May 7-11, 1962.
- D. K. Holmes, J. W. Corbett, R. M. Walker, J. S. Koehler, and Fredrick Seitz
 "On the Interpretation of Radiation Effects in Noble Metals," *Progr. Nucl. Energy, Ser. V*, **3**, 178 (1961).
- D. K. Holmes and D. O. Thompson
 "The Investigation of Radiation Damage Using Mechanical Vibrations," p IX-1 in *Resonance and Relaxation in Metals*, American Society for Metals, 1962.
- L. D. Hulett, Jr.
 "Dissolution of Single Crystals of Copper in Aqueous Systems," presented at Southeastern Section American Chemical Society Meeting, New Orleans, Louisiana, Dec. 7-9, 1961.
- L. H. Jenkins and J. O. Stiegler
 "Electrochemical Dissolution of Single Crystalline Copper," *J. Electrochem. Soc.* **109**, 467 (1962).
- C. E. Klabunde, R. R. Coltman, and D. L. McDonald
 "Low Temperature n, γ Recoil Damage in Cadmium," *Bull. Am. Phys. Soc.* **7**, 171 (1962).
- G. Leibfried
 "Calculation of Averages for Primary Recoil Distributions," *J. Appl. Phys.* **33**, 1933 (1962).
- G. Leibfried and O. S. Oen
 "A Simple Approximation for Classical Scattering at Large Angles," *J. Appl. Phys.* **33**, 2257 (1962).

- E. Lell and R. A. Weeks
 "Dielectric Measurements in Some Irradiated Glasses," presented at Conference on Irradiation Effects in Simple Glasses and Crystals, Rochester, New York, Apr. 12-13, 1962.
- D. I. McDonald, R. R. Coltman, and C. E. Klabunde
 "Contribution of Slow Neutrons to Reactor Damage in Metals," *Bull. Am. Phys. Soc.* **7**, 171 (1962).
- R. B. Murray and A. Meyer
 "Effect of Delta Rays on the Response of Inorganic Scintillators to Heavy Particles," *IRE Trans. Nucl. Sci.* **NS-9**, 33 (1962).
- T. S. Noggle and J. O. Stiegler
 "Fission Fragment Tracks in Metal Films," *J. Appl. Phys.* **33**, 1726 (1962).
 "Fission Fragment Tracks in Thin Films of UO_2 ," pp 47-63 in *Symposium on Radiation Effects in Refractory Fuel Compounds*, ASTM-STP-306, 1961.
- O. S. Oen, M. T. Robinson, and D. K. Holmes
 "The Ranges of Energetic Atoms in Solids. I. Random Models," *Bull. Am. Phys. Soc.* **7**, 171 (1962).
- M. F. Osborne, A. E. Goldman, E. I. Long, Jr., F. R. McQuilkin, and J. G. Morgan
 "Beryllium Sheathed UO_2 Fuel Element Irradiation Tests," *Trans. Am. Nucl. Soc.* **5**, 43 (1962).
- M. F. Osborne and J. G. Morgan
Post-Irradiation Examinations of Uranium Monocarbide, ORNL CF-62-8-15 (Aug. 9, 1962).
- V. K. Paré and D. O. Thompson
 "Temperature Dependence from 250°K to 370°K of Dislocation Pinning in Copper Single Crystals by Radiation Defects," *Acta Met.* **10**, 327 (1962).
- P. E. Reagan
 "Fission Gas Release from Coated Particles Irradiated in ORR Facilities B9 and C1," presented at Third Meeting of Coated Particle Work Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 7-8, 1962.
- L. D. Roberts, H. Pomerance, J. O. Thompson, and C. F. Dam
 "A Mössbauer Study of a Series of Gold Alloys and Compounds," presented at Southeastern Section Meeting, American Physical Society, Tallahassee, Florida, Apr. 5-7, 1962.
- M. T. Robinson, D. K. Holmes, and O. S. Oen
 "Monte Carlo Calculations of the Ranges of Energetic Atoms in Solids," presented at International Colloquium on Ion Bombardment, Bellevue, France, Dec. 4-8, 1961.
 "The Ranges of Energetic Atoms in Solids. II. Lattice Model," *Bull. Am. Phys. Soc.* **7**, 171 (1962).
- H. C. Schweinler
 "Distribution in Kinetic Energy of Daughter Nuclei," p 63 in *Chemical Effects of Nuclear Transformations*, vol I, International Atomic Energy Agency, Vienna, 1961.
- H. C. Schweinler and J. W. Miller
 "Trapping and Recombination Probabilities in Nonmetals," *Bull. Am. Phys. Soc.* **7**, 495 (1962).
- E. Sonder
 "Magnetic Properties of *F* and *M* Centers in Potassium Chloride," *Bull. Am. Phys. Soc.* **7**, 37 (1962).
 "Magnetism of KCl," *Phys. Rev.* **125**, 1203 (1962).
- E. Sonder and S. T. Sekula
 "Effects of Gaseous Impurities on the Magnetic Properties of Copper," *J. Phys. Chem. Solids* **36**, 93 (1962).
- E. Sonder and W. A. Sibley
 "The Relation Between *F* and *M* Centers in KCl at Room Temperature," presented at International Symposium on Color Centers, Stuttgart, Germany, Aug. 21-24, 1962.

- J. O. Stiegler and T. S. Noggle
 "Nitrogen Ion Bombardment of Thin Pt Films," *J. Appl. Phys.* **33**, 1894 (1962).
- D. O. Thompson
 "A Thermally Activated Internal Friction Spectrum in Cu Following Neutron Irradiation," *Acta Met.* **10**, 327 (1962).
- D. O. Thompson and V. K. Paré
 "Effect of Fast Neutron Irradiation on Dislocation Internal Friction in Pure Copper at Megacycle Frequencies," *Bull. Am. Phys. Soc.* **7**, 171 (1962).
 "Measurements of Fast Neutron Produced Defect Motion in Copper from 250°K to 370°K Using Internal Friction Techniques," presented at Diskussionsstagung über Strahlungsschaden und Plastische Verformung, Göttingen, Germany, Oct. 9-13, 1961.
- D. F. Toner
 "LITR Static Capsule Tests for Coated UC_2 Particles," presented at Third Meeting of Coated Particle Work Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 7-8, 1962.
 "Post-Irradiation Examination of MTR-48 Fuel Bodies," presented at Third Meeting of Coated Particle Work Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 7-8, 1962.
- M. S. Wechsler and R. H. Kemohan
 "The Effect of Radiation on Diffusion-Controlled Reactions in Copper-Base Alloys," presented at International Atomic Energy Agency Symposium on Radiation Damage in Solids and Reactor Materials, Venice, Italy, May 7-11, 1962.
- M. S. Wechsler, J. M. Williams, and H. M. Otte
 "The Annealing of Cold-Worked Silicon Bronze," *J. Metals* **14**, 81 (1962).
- R. A. Weeks
 "Paramagnetic Properties of E'_2 Centers in Crystalline Quartz," presented at 1962 Conference on Radiation Effects on Glass, Irradiation Effects in Simple Glasses and Crystals, Rochester, New York, Apr. 12-13, 1962.
 "The Interaction of O-H in Silica and Its Effect on the E_1 Center," presented at Fourteenth Annual Pacific Coast Regional Meeting, American Ceramic Society, San Francisco, California, Oct. 25-27, 1961.
- R. A. Weeks and E. Sonder
 "The Relation Between the Magnetic Susceptibility, Electron Spin Resonance, and the Optical Absorption of the E_1 Center in Fused Silica," presented at First International Conference on Paramagnetic Resonance, Jerusalem, Israel, July 16-20, 1962.
- J. E. White, W. W. Parkinson, D. Binder, and C. D. Bopp
 "A Comparison of Reactor Neutrons to Gamma Radiation in Crosslinking Polystyrene," presented at 2nd Conference on Nuclear Reactor Chemistry, Gatlinburg, Tennessee, Oct. 10-12, 1961.
- M. C. Wittels
 "Stored Energy in the Oak Ridge Graphite Reactor - 1960," *J. Nucl. Energy, Pts. A & B* **15**, 185 (1961).
- M. C. Wittels, J. O. Stiegler, and F. A. Sherrill
 " $Au_{0.82}Hg_{0.18}$: A Transmutation Alloy," *J. Appl. Phys.* **33**, 241 (1962).
 "Radiation Effects in Uranium-Doped Zirconia," *J. Nucl. Energy, Pts. A & B* **16**, 237 (1962).
- F. W. Young, Jr.
 "Elastic-Plastic Transition in Copper Crystals as Determined by an Etch Pit Technique," *J. Appl. Phys.* **32**, 1815 (1961).
 "Etching of Irradiated Copper," *J. Appl. Phys.* **33**, 749 (1962).

F. W. Young, Jr.

"On the Formation of Dislocations Around Precipitate Particles of Cu_2O in Cu," p 103 in *Direct Observation of Imperfections in Crystals* (ed. by J. B. Newkirk and J. H. Wemick), Interscience, New York, 1962.

"On the Growth of Copper Crystals of Low Dislocation Density," *Bull. Am. Phys. Soc.* **7**, 215 (1962).

"On the Yield Stress of Copper Crystals," *J. Appl. Phys.* **33**, 963 (1962).

SOLID STATE SEMINARS AT ORNL

The following speakers gave seminars to members of the Solid State Division during the period covered by this report:

1961

September 12	H. M. Otte, RIAS
"Lattice Parameter Changes Associated with Solute Clustering in Alloys"
September 25	A. A. Johnson, Imperial College, University of London
"Radiation Damage in bcc Transition Metals"
October 10	P. Baruch, Ecole Normale Supérieure, Paris
"Studies of Irradiation Effects in Semiconductors at Ecole Normale Supérieure"
October 19	J. J. Markham, Armour Research Foundation
"The Absorption and Luminescent Properties of the F-Center"
October 26	F. Bassani, Argonne National Laboratory
"Band Structure in Compound Semiconductors"
November 6	G. Watkins, General Electric Research Laboratory
"Radiation-Produced Defects in Silicon"
December 7	R. Smoluchowski, Princeton University
"Present Status of the Mechanism of Defect Formation in Ionic Crystals"
December 14	R. Warren, Westinghouse Research Laboratories
"Purification of Alkali Halides"

1962

February 1	H. J. Stoecker, EURATOM
"Structures of Zirconia Solid Solutions Containing Various Oxides"
February 2	R. K. Swank, University of Illinois
"Lifetimes of Excited States in Color Centers"
February 16	H. Rabin, Naval Research Laboratory
"Equilibria of F and M Centers"
March 14, 15	R. S. Knox, University of Rochester
"Theory of Excitons"
April 6	T. J. Koppenaal, Armour Research Foundation
"The Solid Solution Strengthening in Alpha Cu-Al Single Crystals"
April 18	E. W. J. Mitchell, University of Reading
"Recent Work on Radiation Damage in Solids at Reading University"
June 26	M. M. Saffren, General Electric Research Laboratory
"The Order of Levels of Symmetric Hamiltonians"

- July 12 J. Silcox, Cornell University
 "Dislocations and Magnetic Domain Walls in Thin Films of Cobalt and Nickel"
- August 15 C. Walker, University of Chicago
 "A Neutron Diffraction Study of Short Range Order in Beta-Brass"

During June and July, 1962, W. H. Young of Cornell University gave a series of lectures on "The Theory of Superconductivity."

EDUCATIONAL LECTURES AND SEMINARS

Four speakers from the Solid State Division participated in the University Traveling Lecture Program sponsored by ORINS. They presented the following lectures under the auspices of this program.

J. H. Barrett	University of Texas, October 10, 1961
Rice University, October 11, 1961
A & M College of Texas, October 12, 1961
University of Kentucky, April 6, 1962
"Electrothermal Behavior of Free Electrons in a Magnetic Field"
	University of Cincinnati, April 5, 1962
"Electronic Structure of Solids"
O. L. Curtis, Jr.	University of Texas, April 27, 1962
"Electronic Relaxation Processes in Semiconductors"
W. W. Parkinson	Texas Woman's University, April 8, 1962
A & M College of Texas, May 7, 1962
"The Effects of Radiation on Organic High Polymers"
M. T. Robinson	University of Texas, January 10, 1962
University of St. Thomas, January 12, 1962
University of Houston, January 13, 1962
"Heavy Ion Bombardment of Solids"

Other educational lectures were presented by members of the Division. A series of lectures on "Radiation Damage Research in University Reactors" was given at Georgia Institute of Technology from April 20 to May 25:

D. S. Billington	"Radiation Damage Research at Nuclear Reactors,"
April 20, 1962
J. W. Cleland	"Radiation Damage Investigations on Semiconductors
Using Nuclear Reactors," May 4, 1962
D. K. Holmes	"Theory of the Radiation Damage Process in Solids"
and "Calculation of Fast Neutron Fluxes," May 25,
1962.
D. O. Thompson	"Experiments in Metallic Inelasticity Using Nuclear
Reactors," May 11, 1962
M. C. Wittels	"X-Ray Diffraction Studies of Neutron Irradiated Solids,"
April 13, 1962

The Division also assisted in the program on graduate nuclear engineering education sponsored by General Electric Company, Richland, and Washington State University, held at Richland, Washington, during the month of August:

R. G. Berggren	"Radiation Effects in Carbon Steels and Iron," August 10, 1962
R. M. Carroll	"Fission Gas Release in UO_2 During Irradiation," August 17, 1962
J. H. Crawford, Jr.	"Irradiation Effects in Semiconductors and Ceramics," November 15, 1962
D. K. Holmes	"Radiation Damage Theory," August 1, 1962

H. C. Schweinler was one of the principal lecturers at the ORINS-AEC Summer Institute for Physics Teachers from Small Colleges, held at Oak Ridge July 23 – August 17. He gave a series of 20 lectures on "Quantum Mechanics and Solid State Physics." He also presented a series of five lectures on "Solid State Physics" at a summer school devoted to physics, held at Beloit College, Beloit, Wisconsin, July 11–13, 1962.

A number of other seminars and lectures were presented during the year by members of the Division, both in this country and abroad:

R. M. Carroll	General Electric Company, San Jose, California, May 1, 1962. "Fission Gas Release Studies"
R. R. Coltman	Argonne National Laboratory, Argonne, Illinois, March 1, 1962 "Thermal and Fast Neutron Damage in Metals"
J. H. Crawford, Jr.	Arizona State University, Tempe, Arizona, November 15, 1961 "Radiation Effects in Semiconductors"
O. L. Curtis, Jr.	Arizona State University, Tempe, Arizona, January 8, 1962 Sandia Corporation, Albuquerque, New Mexico, May 2, 1962 "Electronic Relaxation Processes in Semiconductors" Lockheed Missile and Space Corporation, Palo Alto, California, May 15, 1962 "Recombination and Trapping in Co^{60} γ -Irradiated Ge"
G. Leibfried	University of Illinois, Urbana, Illinois, March 2, 1962 Argonne National Laboratory, Argonne, Illinois, March 5, 1962 University of Virginia, Charlottesville, Virginia, March 23, 1962 "Slowing Down of Primary Recoils in Solids"
R. B. Murray	University of Tennessee, Knoxville, Tennessee, December 5, 1961 University of Delaware, Newark, Delaware, June 27, 1962 "Scintillation Process in Alkali Halide Crystals"
O. S. Oen	Neutron Physics Division, ORNL, November 15, 1961 "Monte Carlo Calculations of the Ranges of Energetic Atoms in Solids"

- V. K. Paré CEN, Mol, Belgium, April 24, 1962
"Delayed Pinning Effects in Copper"
- M. T. Robinson The University, Reading, England, December 11, 1961
General Dynamics/Astronautics, San Diego, California,
August 14, 1962
"Heavy Ion Bombardment of Solids"
- H. C. Schweinler Sandia Corporation, Albuquerque, New Mexico, August
22, 1962
"Clean Experiments in Solid State Physics"
- E. Sonder Franklin Institute, Philadelphia, Pennsylvania, Decem-
ber 18, 1961
"Magnetism of Color Centers in Alkali Halides"
- D. O. Thompson Institut fuer Metallphysik, Gottingen, Germany, October
11, 1961
"Radiation Damage Measurements and Defect-Disloca-
tion Interactions in Copper"
- C.C.R. EURATOM, Ispra, Italy, October 26, 1961
"Defect Mobility in Copper from 280°K to 375°K as
Measured by Internal Friction Techniques"
- M. S. Wechsler C.C.R. EURATOM, Ispra, Italy, May 16, 1962
Imperial College, London, England, May 1, 1962
"The Effect of Radiation on Copper-Aluminum Alloys"
- R. A. Weeks Argonne National Laboratory, Argonne, Illinois, October
23, 1961
"The Relationship of the E' Centers and O-H Radicals
in Silica"
- Series of lectures at Summer School sponsored by NATO
and Greek AEC, Rhodes, Greece, July 12-13, 1962
"The Structure of Paramagnetic Defects in Crystals"
- M. C. Wittels Argonne National Laboratory, Argonne, Illinois, April
13, 1962
"Some X-Ray Diffraction and Thermal Studies of
Radiation Damage"
- F. W. Young, Jr. Yale University, New Haven, Connecticut, November
9, 1961
University of Virginia, Charlottesville, Virginia,
November 10, 1961
Technische Hochschule, Stuttgart, Germany, July 6,
1962
"On the Yield Stress of Copper"
- Ford Motor Company, Detroit, Michigan, January 24,
1962
Michigan State University, East Lansing, Michigan,
January 26, 1962
"Mechanical Properties of Dislocations in Copper"
- University of Illinois, Urbana, Illinois, May 25, 1962
"Deformation of Copper Crystals"
- Institute for Physical Chemistry, Acad. Nauk, Moscow,
USSR, July 6, 1962
"Crystal Imperfections and Chemical Reactivity"

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INTRODUCTION

The ORNL-3364 Key Word Index provides an insight to the contents of the *Solid State Division Annual Progress Report for the Period Ending August 31, 1962* (ORNL-3364). The Index contains four parts: (1) Key Word Index, (2) Title Index, (3) Publications, and (4) Author Index.

The Key Word Index section is a combined alphabetical listing of the Title Index and the Publications. If it is necessary to know a complete title, use the identification number and refer to the Title Index or the Publications section.

The Title Index is a listing of chapter titles and their primary subordinate headings, with the addition of key words to further aid in the understanding of the work reported. An example of the numbering system is 62-III-11-2, where 62 indicates the year of publication, III is the part number (or major division), 11 is the chapter, and 2 is the number of the primary heading within the chapter. Example:

The Publications section is a listing of published work by Division members for the period August 31, 1961, to August 31, 1962. (Papers presented at technical meetings and articles submitted for publication are not included.) An example of the numbering system is 62-PUB-42, where 62 shows the year in which the publication was first reported as such, PUB identifies the work as having been published, and the last number was assigned to clearly identify a specific publication. Example:

The Author Index is an alphabetical listing of the names of authors, followed by the numbering systems used in the Title Index and the Publications. This list shows reported work to which each author contributed.

To locate specific work in the Annual Report, it is necessary to use only the last two groups of numbers, since chapters are numbered consecutively.

The computer used for this program does not permit the use of lower-case type, superscript and subscript numbers, and certain specific punctuation marks. Therefore, to avoid confusion, Greek letters, chemical symbols, formulas, and names of elements and compounds are written out. When a title is too long to be given on one line, a slant line (/) indicates the title break.

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- 62-VI-23 RADIATION DOSIMETRY IRRADIATION PRODUCED ACID CONCENTRATIONS OF TETRACHLOROETHANE AND DECANE IN A
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BOPP, C. D. + SISMAN, O. + KIRKLAND, W. K.
PARKINSON, W. W.

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PUBLICATIONS

- 62-PUB-1 RADIATION EFFECTS ON DEEP-LEVEL IMPURITY STATES IN GERMANIUM
BASS, R. F. + CLELAND, J. W. + CRAWFORD, J. H., JR.
BULL. AM. PHYS. SOC. 6, 461 (1961)
- 62-PUB-2 THE DEPENDENCE OF RADIATION-ENHANCED DIFFUSION IN COPPER - ALUMINUM ON NEUTRON FLUX AND ENERGY SPECTRUM
BASSANI, C. + WECHSLER, M. S. + WILLIAMS, J. M.
BARRETT, J. H. + KERNOHAN, R. H.
BULL. AM. PHYS. SOC. 7, 170 (1962)
- 62-PUB-3 CRYOGENIC TECHNIQUES FOR IRRADIATION STUDIES IN NUCLEAR REACTORS
BLEWITT, T. H. + COLTMAN, R. R.
P 274 IN EXPERIMENTAL CRYOPHYSICS (ED. BY F. E. HOARE ET AL.),
BUTTERWORTHS, LONDON, 1961
- 62-PUB-4 LOW TEMPERATURE RESISTIVITY OF SODIUM AND THE EFFECTS OF NUCLEAR IRRADIATION
BLEWITT, T. H. + COLTMAN, R. R. + KLABUNDE, C. E.
MCDONALD, D. L.
J. APPL. PHYS. 33, 1612 (1962)
- 62-PUB-5 CALORIMETRIC MEASUREMENT OF NUCLEAR HEATING IN A REACTOR
BOPP, C. D. + TOWNS, R. L.
NUCL. SCI. ENG. 13, 245 (1962)
- 62-PUB-6 MAGNETIC SUSCEPTIBILITY OF POTASSIUM HEXACHLORORHENATE(IV) AND POTASSIUM HEXABROMORHENATE(IV) FROM 5K TO 300K
BUSEY, R. H. + SONDER, E.
J. CHEM. PHYS. 36, 93 (1962)
- 62-PUB-7 ARGON ACTIVATION MEASURES IRRADIATION FLUX CONTINUOUSLY
CARROLL, R. M.
NUCLEONICS 20(2), 42 (1962)
- 62-PUB-8 CONTINUOUS RELEASE OF FISSION GAS FROM URANIUM-DIOXIDE DURING IRRADIATION
CARROLL, R. M.
P 111 IN SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS, ASTM-STP-306, 1961
- 62-PUB-9 SPIN-LATTICE RELAXATION OF E-PRIME-ONE CENTERS IN SILICON-DIOXIDE
CASTLE, J. G., JR. + FELDMAN, D. W. + WEEKS, R. A.
BULL. AM. PHYS. SOC. 7, 188 (1962)
- 62-PUB-10 THE NATURE AND YIELD OF RADIATION-INDUCED DEFECTS IN SEMICONDUCTORS
CLELAND, J. W. + BASS, R. F. + CRAWFORD, J. H., JR.
BULL. AM. PHYS. SOC. 7, 186 (1962)
- 62-PUB-11 OPERATION OF RESEARCH MATERIALS INFORMATION CENTER
CONNOLLY, T. F.
ORNL TM-295 (JULY 30, 1962)
- 62-PUB-12 RADIATION STABILITY OF NONMETALLIC STRUCTURES
CRAWFORD, J. H., JR.
P 371 IN PROGRESS IN NUCLEAR ENERGY, (ED. BY H. M. FINNISTON AND J. P. HOWE), SERIES V, VOL 3, PERGAMON PRESS, NEW YORK, 1961
- 62-PUB-13 TRANSMUTATION DOPING AND RECOIL EFFECTS IN SEMICONDUCTORS EXPOSED TO THERMAL NEUTRONS
CRAWFORD, J. H., JR. + CLELAND, J. W.
P 269 IN RADIOISOTOPES IN THE PHYSICAL SCIENCES AND INDUSTRY,
INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, 1962
- 62-PUB-14 EFFECT OF A PHASE TRANSFORMATION ON THE VAPOR PHASE GROWTH OF SINGLE-CRYSTAL MERCURIC-SULFIDE
CURTIS, O. L., JR.
J. APPL. PHYS. 33, 2461 (1962)
- 62-PUB-15 CARRIER RECOMBINATION AND TRAPPING PROCESSES IN IRRADIATED GERMANIUM
CURTIS, O. L., JR. + CRAWFORD, J. H., JR.
DISCUSSIONS FARADAY SOC. 31, 107 (1961)
- 62-PUB-16 RADIATION-INDUCED RECOMBINATION AND TRAPPING CENTERS IN GERMANIUM I. THE NATURE OF THE RECOMBINATION PROCESS
CURTIS, O. L., JR. + CRAWFORD, J. H., JR.
PHYS. REV. 124, 1731 (1961)
- 62-PUB-17 RADIATION-INDUCED RECOMBINATION AND TRAPPING CENTERS IN GERMANIUM II. ANNEALING IN GAMMA-IRRADIATED, ANTIMONY-DOPED AND ARSENIC-DOPED MATERIAL
CURTIS, O. L., JR. + CRAWFORD, J. H., JR.
PHYS. REV. 126, 1342 (1962)
- 62-PUB-18 STUDIES OF THE SCINTILLATION PROCESS IN THALLIUM-ACTIVATED CESIUM-IODIDE
GWIN, R. + MURRAY, R. B.
IRE TRANS. NUCL. SCI. NS-9, 28 (1962)
- 62-PUB-19 APPROXIMATE METHODS FOR TREATING ANNEALING PROCESSES WHICH INVOLVE A SPECTRUM OF ACTIVATION ENERGIES
HOLMES, D. K.
P 449 IN CHEMICAL EFFECTS OF NUCLEAR TRANSFORMATIONS, VOL I,
INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, 1961
- 62-PUB-20 ON THE INTERPRETATION OF RADIATION EFFECTS IN NOBLE METALS
HOLMES, D. K. + CORBEIL, J. W. + WALKER, R. M.
KOEHLER, J. S. + SEITZ, F.
P 178 IN PROGRESS IN NUCLEAR ENERGY, (ED. BY H. M. FINNISTON AND J. P. HOWE), SERIES V, VOL 3, PERGAMON PRESS, NEW YORK, 1961

- 62-PUB-21 THE INVESTIGATION OF RADIATION DAMAGE USING MECHANICAL VIBRATIONS
HOLMES, D. K. + THOMPSON, D. O.
P IX-1 IN RESONANCE AND RELAXATION IN METALS, AMERICAN SOCIETY FOR
METALS, 1962
- 62-PUB-22 ELECTROCHEMICAL DISSOLUTION OF SINGLE CRYSTALLINE COPPER
JENKINS, L. H. + STIEGLER, J. O.
J. ELECTROCHEM. SOC. 109, 467 (1962)
- 62-PUB-23 LOW TEMPERATURE NEUTRON, GAMMA RECOIL DAMAGE IN CADMIUM
KLABUNDE, C. E. + COLTMAN, R. R. + McDONALD, D. L.
BULL. AM. PHYS. SOC. 7, 171 (1962)
- 62-PUB-24 CALCULATION OF AVERAGES FOR PRIMARY RECOIL DISTRIBUTIONS
LEIBFRIED, G.
J. APPL. PHYS. 33, 1933 (1962)
- 62-PUB-25 A SIMPLE APPROXIMATION FOR CLASSICAL SCATTERING AT LARGE ANGLES
LEIBFRIED, G. + OEN, O. S.
J. APPL. PHYS. 33, 2257 (1962)
- 62-PUB-26 CONTRIBUTION OF SLOW NEUTRONS TO REACTOR DAMAGE IN METALS
McDONALD, D. L. + COLTMAN, R. R. + KLABUNDE, C. E.
BULL. AM. PHYS. SOC. 7, 171 (1962)
- 62-PUB-27 EFFECT OF DELTA RAYS ON THE RESPONSE OF INORGANIC SCINTILLATORS TO HEAVY PARTICLES
MURRAY, R. B. + MEYER, A.
IRE TRANS. NUCL. SCI. NS-9, 33 (1962)
- 62-PUB-28 FISSION FRAGMENT TRACKS IN METAL FILMS
NOGGLE, T. S. + STIEGLER, J. O.
J. APPL. PHYS. 33, 1726 (1962)
- 62-PUB-29 FISSION FRAGMENT TRACKS IN THIN FILMS OF URANIUM-DIOXIDE
NOGGLE, T. S. + STIEGLER, J. O.
PP 47 - 63 IN SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL
COMPOUNDS, ASTM-STP-306, 1961
- 62-PUB-30 THE RANGES OF ENERGETIC ATOMS IN SOLIDS I. RANDOM MODEL
OEN, O. S. + ROBINSON, M. T. + HOLMES, D. K.
BULL. AM. PHYS. SOC. 7, 171 (1962)
- 62-PUB-31 BERYLLIUM SHEATHED URANIUM-DIOXIDE FUEL ELEMENT IRRADIATION TESTS
OSBORNE, M. F. + GOLDMAN, A. E. + LONG, E. L., JR.
MCQUILKIN, F. R. + MORGAN, J. G.
TRANS. AM. NUCL. SOC. 5, 43 (1962)
- 62-PUB-32 POST-IRRADIATION EXAMINATIONS OF URANIUM MONOCARBIDE
OSBORNE, M. F. + MORGAN, J. G.
ORNL CF-62-8-15 (AUG. 9, 1962)
- 62-PUB-33 TEMPERATURE DEPENDENCE FROM 250K TO 370K OF DISLOCATION PINNING IN COPPER SINGLE CRYSTALS BY
RADIATION DEFECTS
PARE, V. K. + THOMPSON, D. O.
ACTA MET. 10, 327 (1962)
- 62-PUB-34 THE RANGES OF ENERGETIC ATOMS IN SOLIDS II. LATTICE MODEL
ROBINSON, M. T. + HOLMES, D. K. + OEN, O. S.
BULL. AM. PHYS. SOC. 7, 171 (1962)
- 62-PUB-35 DISTRIBUTION IN KINETIC ENERGY OF DAUGHTER NUCLEI
SCHWEINLER, H. C.
P 63 IN CHEMICAL EFFECTS OF NUCLEAR TRANSFORMATIONS, VOL I,
INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, 1961
- 62-PUB-36 TRAPPING AND RECOMBINATION PROBABILITIES IN NONMETALS
SCHWEINLER, H. C. + MILLER, J. W.
BULL. AM. PHYS. SOC. 7, 495 (1962)
- 62-PUB-37 MAGNETIC PROPERTIES OF F-CENTERS AND M-CENTERS IN POTASSIUM-CHLORIDE
SONDER, E.
BULL. AM. PHYS. SOC. 7, 37 (1962)
- 62-PUB-38 MAGNETISM OF POTASSIUM-CHLORIDE
SONDER, E.
PHYS. REV. 125, 1203 (1962)
- 62-PUB-39 EFFECTS OF GASEOUS IMPURITIES ON THE MAGNETIC PROPERTIES OF COPPER
SONDER, E. + SEKULA, S. T.
J. PHYS. CHEM. SOLIDS 36, 93 (1962)
- 62-PUB-40 NITROGEN ION BOMBARDMENT OF THIN PLATINUM FILMS
STIEGLER, J. O. + NOGGLE, T. S.
J. APPL. PHYS. 33, 1894 (1962)
- 62-PUB-41 A THERMALLY ACTIVATED INTERNAL FRICTION SPECTRUM IN COPPER FOLLOWING NEUTRON IRRADIATION
THOMPSON, D. O.
ACTA MET. 10, 327 (1962)
- 62-PUB-42 EFFECT OF FAST NEUTRON IRRADIATION ON DISLOCATION INTERNAL FRICTION IN PURE COPPER AT MEGACYCLE
FREQUENCIES
THOMPSON, D. O. + PARE, V. K.
BULL. AM. PHYS. SOC. 7, 171 (1962)

- 62-PUB-43 THE ANNEALING OF COLD-WORKED SILICON BRONZE
WECHSLER, M. S. + WILLIAMS, J. M. + OTTE, H. M.
J. METALS 14, 81 (1962)
- 62-PUB-44 STORED ENERGY IN THE OAK-RIDGE GRAPHITE REACTOR - 1960
WITTELS, M. C.
J. NUCL. ENERGY, PTS. A AND B 15, 185 (1961)
- 62-PUB-45 GOLD(0.82) - MERCURY(0.18) - A TRANSMUTATION ALLOY
WITTELS, M. C. + STIEGLER, J. O. + SHERRILL, F. A.
J. APPL. PHYS. 33, 241 (1962)
- 62-PUB-46 RADIATION EFFECTS IN URANIUM-DOPED ZIRCONIA
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J. NUCL. ENERGY, PTS. A AND B 16, 237 (1962)
- 62-PUB-47 ELASTIC-PLASTIC TRANSITION IN COPPER CRYSTALS AS DETERMINED BY AN ETCH PIT TECHNIQUE
YOUNG, F. W., JR.
J. APPL. PHYS. 32, 1815 (1961)
- 62-PUB-48 ETCHING OF IRRADIATED COPPER
YOUNG, F. W., JR.
J. APPL. PHYS. 33, 749 (1962)
- 62-PUB-49 ON THE FORMATION OF DISLOCATIONS AROUND PRECIPITATE PARTICLES OF CUPROUS-OXIDE IN COPPER
YOUNG, F. W., JR.
P 103 IN DIRECT OBSERVATION OF IMPERFECTIONS IN CRYSTALS (ED. BY
J. B. NEWKIRK AND J. H. WERNICK), INTERSCIENCE, NEW YORK, 1962
- 62-PUB-50 ON THE GROWTH OF COPPER CRYSTALS OF LOW DISLOCATION DENSITY
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BULL. AM. PHYS. SOC. 7, 215 (1962)
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