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TO : R. L. Metter, Chief, Production Control Section, DATE: March 20, 1953
Technical Information Service, Oak Ridge

FROM : D. W. Lillie, Chief, Metallurgy & Materials *DWL*
Branch, Division of Research, Washington

SUBJECT: REPORT COO-102

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Also attached is a copy of a memo to Dr. Whaley indicating that COO-103 is also approved. This report was reviewed on the basis of a carbon copy submitted to us by Purdue and I believe the original will be sent to you from Purdue for duplication and distribution.

enclosure:
Report COO-102

cc: R. M. Whaley

ENCLOSURES CHECKED

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Date January 14, 1953

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Author: _____

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FIRST QUARTERLY REPORT ON RADIATION DAMAGE RESEARCH
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repro cy

Progress is reported on the hardness of Mo before and after irradiation, the influence of neutrons on the low-temperature atomic heat of Si, an x-ray and electron diffraction study of radiation damage in single crystals of Ge and Si, thermal conductivity of Ge, electric measurement^s on Mo, and electron bombardment of Ge.

~~(L.M.T.)~~ (L.T.W.)

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N-8

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COO-102

RADIATION DAMAGE RESEARCH

First Quarterly Report — July 1 to September 30;
1952

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and
Purdue Research Foundation
Lafayette, Indiana

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I. HARDNESS MEASUREMENTS OF MOLYBDENUM BEFORE AND AFTER BOMBARDMENT

By T. C. Harmon, R. Eidam and I. G. Geib

In the work reported previously on the hardness of Mo sheet, the hardness numbers were obtained using a Tukon tester equipped with a Knoop indenter. To study deviations in hardness number, measurements of micro-hardness within a grain and at a grain boundary are being made with a Bergsman microhardness tester attached to a Vickers projection microscope.

The hardness of cold rolled and annealed Mo sheets has been measured before and after bombardment with 8.4 to 10 Mev deuterons from the cyclotron. The annealed sheets were obtained by heating for 20 hours at 2400°F in dry hydrogen. During bombardment part of the specimen was shielded from the beam so that neighboring sections, shielded and unshielded regions, could be compared to eliminate as far as possible the effects of surface conditions.*

The sheets were hand-polished, electropolished and etched. After etching, the grain boundaries of the annealed material are clearly defined. The heat treatment used gives grain growth and a wide range of grain sizes. In one annealed sample, the grain size varied from $4 \times 5 \mu$ to $144 \times 360 \mu$. In measuring the annealed samples, indentations were made in large grains so that an adequate number of indentations in a single grain could be obtained.

Factors influencing hardness numbers obtained with the Bergsman tester were investigated. The Vickers microscope has been mounted on rubber to isolate it from vibration. Hardness number was determined as a function of time of application of load for loads of 25, 50, and 100 gm. Smooth curves were obtained with hardness number decreasing with increased load and increased times of application. Measurements have been made with a 25 gm load applied for 15 sec, with a 50 gm load applied for 50 sec. and with a 50 gm load applied for 120 sec. In agreement with the result to be expected from the shape of the hardness-time of loading curves, the latter conditions give more uniform results. The influence of the rate of descent of the sample was studied within the range from 0.5 to 2mm/min. with a 50 gm load applied for 50 sec. The hardness number does not vary more than ± 2 within this range. Hardness numbers given in this report were obtained with a rate of descent of ~ 1 mm/min. A change of approximately 15 V.P.N. (Vickers Pyramidal Number) can be observed within a grain by rotating the grain through

* This technique was used successfully sometime ago by K. Lark-Horovitz in the hardness testing of deuteron bombarded Cu-Be alloys.

an angle of 45° . This may be a result of the orientation of the grains in the sheet. This variation has no effect on comparisons made of hardness before and after bombardment, for the sheet is always placed in the specimen holder orientated in the same way with respect to the indenter.

With a load of 50 gm applied for 120 sec, the variation in hardness from grain to grain in the same sample can be at least as much as 20 V.P.N. For all the annealed samples measured, the range of hardness is from 169 to 206 V.P.N. under these conditions.

The original thicknesses of the sheets were 5 mil or 10 mil. Surface preparation reduced the thickness of the 5 mil to 3 mil and the 10 mil to 7 mil. The samples were cooled with dry ice and alcohol during bombardment and were kept at room temperature until the radioactivity decayed sufficiently for safe processing.

A cold rolled specimen, H-4, of 3 mil thickness was given 13.8μ amp-hr/cm² irradiation. The bombarded region was discolored and the sample was cleaned before measurement. With the cold rolled specimens, individual grains cannot be distinguished. Indentations were made at equally spaced intervals across parts of the bombarded and shielded regions with a 25 gm load applied for 15 sec, 12 days after bombardment; 12 readings across the bombarded region gave from 227 to 270 with an average hardness of 256 V.P.N.; 12 readings across the shielded region gave from 246 to 270 with an average hardness of 259 V.P.N. On the average, there was no difference between the bombarded and shielded regions in hardness.

An annealed specimen, H-6, of 3 mil thickness was given 13.5μ amp-hr/cm² irradiation. The bombarded region was discolored and the sample was cleaned after bombardment. Three readings in each of 4 grains in the bombarded region and each of 4 grains in the shielded region were made using a 25 gm load applied for 15 sec., and the hardness number compared with values obtained for the same grains before irradiation. Average hardness numbers are given in Table I. The hardness numbers of the grains in the bombarded region are greater by 28 to 30% after irradiation but the hardness numbers of the shielded grains are also greater, although the percentage changes are smaller 6, 8, 20, and 8%. The specimen was allowed to stand an additional 6 days and remeasured with the results given in column 3. Within the estimated error, ± 10 , the values are the same as those in column 2. This sample was given an additional irradiation of 13.8μ amp-hr/cm² irradiation. The bombarded region was discolored and it was cleaned before measurement. The values in column 4 agree with those in column 3 indicating that the added irradiation had produced no change in hardness, although the values in column 3 are not as high as the average hardness of cold rolled sheet.

An annealed specimen, H-7, of 3 mil thickness was given 13.4μ amp-hr/cm² irradiation. The bombarded region was discolored and the sample was cleaned after bombardment. The sample was measured in the same way as H-6. Average hardness numbers are given in Table II. In agreement with the results for H-6,

TABLE I.

Hardness of annealed Molybdenum, V. P. N., specimen H-6.

	1. Before Irradiation	2. 4 days after 13.5μ amp-hr/cm ²	3. 10 days after 13.5μ amp-hr/cm ²	4. 4 days after added 13.8μ amp-hr/cm ²
Bombarded Region				
Grain 1	174	224	228	220
2	186	241	236	236
3	188	241	233	242
4	187	232	237	227
Shielded Region				
Grain 1	185	197	201	198
2	180	195	196	188
3	169	202	197	197
4	174	188	179	188

4

TABLE II.

Hardness of annealed Molybdenum, V.P.N., specimen H-7

	1. Before Bombardment	2. 5 days after 13.4 μ amp-hr/cm ²	3. 12 days after 13.4 μ amp-hr/cm ²
Bombardment Region			
Grain 1	204	273	285
2	171	300	280
3	190	252	275
4	203	274	279
Shielded Region			
Grain 1	208	208	212
2	213	207	213
3	194	197	206
4	220	218	215

the grains in the bombarded region are harder after irradiation. The hardness number of grains 1, 3, and 4 are greater by 34%, 33%, and 35% respectively but the number of grain 2 is higher by an abnormal 76%. Furthermore, three of the grains have hardness numbers higher than the highest value 270 V.P.N. recorded for cold rolled H-4. The specimen was remeasured 12 days after bombardment and three of the deviations + 12, -20 and + 23 are higher than the estimated error in measurement. It may be that the high values and the high deviations are a result of inadequate cleaning, although the grains appeared clean when viewed with the microscope. This sample was irradiated for an additional 14.2μ amp-hr/cm². It was stained and this stain could not be removed. No measurements were made after the added irradiation.

In general, the results on the molybdenum samples H-4, H-6 and H-7 agree with the results reported previously⁽¹⁾. The hardness of annealed Mo (H-6, H-7) is increased by deuteron irradiation and the hardness of cold rolled Mo(H-4) is not changed. An increase in hardness may be attributed to the influence of bombardment produced defects on the elastic properties. All samples were exposed and treated in the same way. However hardness tests before and after irradiation reveal some striking differences.

Using average values for a discussion of the results, the hardness of H-6 was increased from 184 to 234 (V.P.N.) by 13.5μ amp-hr/cm² irradiation. The hardness of the shielded region of H-6 increased from 177 to 196. (Table I). The hardness, 234, is less than the hardness of the cold rolled H-4, 258. On the other hand, the hardness of H-7 was increased from 192 to 275 by 13.4μ amp-hr/cm² irradiation, being greater than the hardness of the cold rolled H-4, while the hardness of the shielded region remained the same.(Table II).

Although cold work hardening and radiation hardening are attributed to lattice disturbances, the types of disturbances are probably different. To determine if the hardness could be increased by further irradiation and to see if the hardness of H-6 could be made greater than that of the cold rolled sample, H-6 and H-7 were given additional irradiation. H-6 was measured after removal of the brown stain produced during bombardment. The hardness after the irradiation, 231, is the same as before 234.

In evaluating these results, the appearance of a stain on the surface must be considered. This brown stain is usually observed only on the bombarded part of the sample. Generally, the bombarded region is marked clearly by the stain which appears on the back of the sample as well as on the front, although it is not as pronounced on the back. The region around the bombarded section which is exposed to the same atmosphere appears clean. The stain appears to be a result of the formation of the thin film on the surface. Several impressions were made

(1) I. G. Geib and R. E. Grace, Progress Report June 1, 1952.

through the stain with the hardness indenter. Examination of an indentation with the Vickers microscope showed that the film cracks around the indentation. X-ray diffraction patterns of the bombarded and unbombarded regions of an irradiated sample were recorded by transmission and back reflection. No differences were found in the patterns from bombarded and unbombarded regions.

The cause and nature of the film are yet unknown. It can be removed by wiping with cotton dipped in trichloroethylene or acetone, trichloroethylene being more effective, but the process is slow. One to two hours are required to clean a sample. After a final cleaning with methyl alcohol, the sample appears clean when examined with the Vickers microscope. The grain boundaries are plain and there is no difficulty measuring a grain which was measured before irradiation. The limited information given by the cleaning process suggests that the film is a carbon compound.

Although the sample appears clean after the removal of the film, it is not certain that the surfaces of the sample are the same before and after irradiation. Carbide or oxide formation may exist, the presence of which could affect the micro hardness measurements. If a compound is formed, the measured hardness may be a function of the thickness and a thin layer may be protection against further attacks. Surface effects may be the explanation for the difference between H-6 and H-7 in hardness after irradiation and of the fact that the hardness of H-6 was not increased by further irradiation beyond 13.5μ amp-hr/cm². Further experiments are needed on the influence of surface layers on the hardness measurements.

In view of the variations encountered with H-6 and H-7, measurements are now being made with a 50 gm load applied for 120 sec. Using an annealed sample, the hardness at the center of a grain has been compared with the hardness at a point midway between the center and the grain boundary. Twenty-six measurements gave maximum deviations + 5.2 and -5.5 with no significant difference between the points measured.

Work is now in progress on one cold rolled and two annealed specimens which are to be given much smaller irradiations to keep the discoloration to a minimum. Each has been given an irradiation of 1.9μ amp-hr/cm². The cold rolled, H-15 and one annealed specimen, H-8, of thickness 7 mil appear clean. The other annealed specimen, H-14, of 7 mil thickness, is slightly stained.

Measurements are being made on the annealed 10 mil Mo sheets reduced to 7 mil thickness by the surface preparation to compare the hardness at the grain boundary with the hardness in the interior of the grain. With a 50 gm load and 120 sec. time of loading, there is very little difference between the hardness measured when the indenter strikes a boundary and the hardness measured in the adjacent grains. In a set of twenty measurements it was found that in nine of them the hardness at the boundary was greater than that of either bordering grain. In eight the hardness number for the boundary was between the numbers for the adjacent grains, and in three the hardness of the boundary was less than that

of the adjacent grains. On the average the hardness at a grain boundary is ~ 1.5 V.P.N. greater than the grain interior hardness. In a typical set of values, one grain interior had a hardness of 204.2, the grain boundary 204.6 and 205.8 and the other grain interior 203.8 V.P.N. It is proposed that the boundary hardness be studied as a function of load for loads less than 50 gm.

All measurements on Mo sheets have been made at room temperature and all samples have been kept at room temperature except during bombardment. It is proposed that samples be kept at dry ice temperature and that consideration be given to making measurements at this temperature.

II. THE INFLUENCE OF NEUTRON BOMBARDMENT ON THE LOW TEMPERATURE ATOMIC HEAT OF SILICON

By P. H. Keesom, K. Lark-Horovitz and N. Pearlman*

Experiments at Purdue with 10 Mev deuterons and at Oak Ridge with fast neutrons have indicated that irradiation produces both electron and hole traps in silicon, since both N- and P-type material become very poorly conducting (resistivities as high as 250,000 ohm-cm have been reached in this way). Thus a result of the lattice disorder (vacant sites and interstitial atoms) produced by elastic collisions is the removal of free carriers. (1)(2). Since annealing at 450°C restores the initial conductivity, it appears to remove the electron and hole traps and release the trapped carriers. Hence any electronic (or hole) contribution to the atomic heat at very low temperatures should be decreased by bombardment and then restored by annealing at elevated temperatures.

The lattice contribution to the atomic heat at very low temperatures should also be sensitive to bombardment because of the dependence of the thermal vibrations on the interatomic forces, which in turn are affected by the disordering of the lattice (3)(4)(5). This has been known for some time from the investigation of the specific heat of the so-called metamict crystals (6), in which the bombardment is produced by radioactive inclusions in the lattice.

In order to see if such changes could be observed experimentally, the atomic heat of two silicon ingots, both kindly supplied by Dr. G. L. Pearson of the Bell Telephone Laboratories, have been measured before and after exposure to about $10^{18}/\text{cm}^3$ neutrons in the nuclear reactor at Oak Ridge. Each ingot weighed about 260 grams. SiV had been doped with 0.01% (by weight) of boron, while the impurity concentration of SiII was not known. SiII was then annealed at several temperatures up to 780°C (See Table I. .), being held at each temperature for the time indicated, and then cooled at about 10°C/hour. Its heat capacity below 5°C was measured after each heat treatment. Annealing of SiV is under way at present.

The experimental method was similar to that of Nernst and Eucken (7) and has been described elsewhere (8). At very low temperatures, the contribution of the lattice to the atomic heat will be given by (9)

$$1944 (T/\Theta)^3 = \alpha T^3 \text{ joules/mole degree} \quad (1)$$

*Assisted by Signal Corps contract, and A.E.C. contract.

TABLE I.

Lattice and Carrier Contributions to Atomic Heat of Silicon as a Function of Bombardment and Heat Treatment.

Run		N	Θ	γ - joules/mol deg ²
1	Si II-before bombardment	18	645°K	13.03 x 10 ⁻⁶
2	Si II-before bombardment	33	659	22.51
3	Si II-before bombardment	35	661	22.51
	Runs 1, 2 and 3 combined	86	658	21.02
4	Si IIB-after bombardment	18	633	1.11
5	Si IIB-after bombardment	20	640	1.96
	Runs 4 and 5 combined	38	637	1.55
6	Si IIB(A1)-anneal at 135°C-24 hrs.	15	628	-0.91
7	Si IIB(A2)- " " 253°C-24 hrs.	17	632	0.43
8	Si IIB(A3)- " " 472°C-24 hrs.	16	628	-2.08
9	Si IIB(A4)- " " 455°C-48 hrs.	13	637	1.74
	Runs 6, 7, 8 and 9 combined	61	632	-0.03
10	Si IIB(A5)-anneal at 780°C-24 hrs.	14	642	13.69
11	Si V - before bombardment	39	658	34.61
12	Si V - after bombardment	17	636	9.26

where Θ is the Debye temperature. The contribution of the carriers can be written⁽¹⁰⁾

$$1.62 \times 10^{-22} V n^{1/3} \mu^{-1} T = \gamma T \text{ joules/mole degree} \quad (2)$$

where V is the atomic volume, n is the carrier concentration per cm^3 , and μ is the ratio of carrier mass to electron mass. Other evidence indicates that this ratio is about unity for carriers in silicon⁽¹¹⁾. Hence the total atomic heat should have the form

$$C_V = \alpha T^3 + \gamma T \quad (3)$$

at very low temperatures. We verified this form of temperature dependence for our data by plotting C_V/T vs T^2 and observed that the points scattered about a straight line for each run (See Figures 1 and 2). The slope α and intercept γ were calculated by least squares, and Θ (calculated from α using Eq. (1)) and γ are listed in Table I. The number of heat capacity measurements made during each run is given in the third column of Table I.

The electronic term in the atomic heat of SiII became very small after bombardment, and remained small after the first four anneals. There would appear to be no significant change in the electronic term after any of these treatments, while the last anneal restores the original linear term, within the accuracy of our measurements. We attempted to determine n , the carrier concentration, independently from measurements of the Hall constant, (performed by D. Finlayson) but the agreement found with n determined from Eq. (2) (about 10^{18} before bombardment) is inconclusive since probe measurements of the large ingot showed it to be very inhomogeneous, with both N- and P-type regions of high and low conductivity. SiV is more homogeneous, apparently being P-type throughout. The Hall constant was measured in the range 4-300°K by H. Fritzsche and found to be roughly constant at a value corresponding to a hole concentration of 10^{19} cm^{-3} , which is also the value to be expected from the amount of boron added to the melt. This hole concentration and the observed value of γ lead to the value 0.8 for μ (see Eq. (2)). After bombardment, the decreased value of γ indicates that about 3/4 of the holes have been trapped (if μ has not changed). This leads to the estimate that for each incident neutron, about 7 traps are produced. Hall constant and resistivity are now being measured on SiVB.

The effect on the Debye temperature is not as clear, although it also appears to decrease after bombardment, and to rise after the last heat treatment. The initial decrease would imply that the decrease of elastic constants due to vacancies predominates over the increase due to interstitial atoms. Dienes' calculations for copper and sodium show that this situation would be

Fig. 1—Plot of C/T vs T^2 for silicon sample SiII before and after neutron bombardment and annealing.

Fig. 2—Plot of C/T vs T^2 for boron-doped silicon sample SiV before and after neutron bombardment.

favorable in an open lattice such as the diamond structure in which silicon crystallizes.

Our results thus lend further support to the idea that lattice disorder produced by neutron bombardment results in carrier traps in silicon which can be healed out by suitable heat treatment. It also confirms the division of the total atomic heat into two parts as indicated by Eq. (3) and the identification of the linear term with the contribution of the carriers.

We would like to thank L. Roth for the probe measurements and heat treatment.

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III. AN X-RAY AND ELECTRON DIFFRACTION STUDY OF RADIATION DAMAGE IN SINGLE CRYSTALS OF GERMANIUM AND SILICON

By W. P. Binnie and A. M. Liebschutz

It was suggested by K. Lark-Horovitz that the extrastructural scattering be investigated as a function of nucleon irradiation. Measurements of the Bragg reflection give an integrated effect and therefore do not readily detect changes in the Bragg intensity, but measurements of scattering within the diffuse domain surrounding a reciprocal lattice point make this detection possible.

This latter procedure can only be carried out accurately by using a narrow parallel beam of strictly monochromatic X-radiation and a stationary single crystal. Standard monochromating crystals are NaCl and CaF_2 , the former giving a ratio of the intensities of the first order to the second order of approximately 20:1 and the latter a ratio of 600:1. Therefore, the reflected radiation from CaF_2 has been used throughout this study of diffuse scattering to ensure the minimum percentage of the second order in the collimated X-ray beam. A high intensity source of molybdenum K α radiation was obtained from the X-ray tube mentioned in last year's report. However, in order to reduce the time of its construction, this tube was built with parts of an older model which have proved unsatisfactory for continuous running during long exposures. Therefore, a new filament assembly has been made which provides an adjustment for the focal spot size in order to obtain the optimum intensity. The auto-transformer with its inconvenient taps has been replaced by a powerstat and a completely new tube body is being installed to ensure better cooling.

In order to scan a diffuse domain thoroughly, three degrees of movement are required by the crystal and/or the recording device. This is not possible with the present equipment, but, nevertheless, a preliminary photographic survey has been made of selected diffuse domains of germanium with a rotational movement being given to the crystal. Three germanium single crystals ($3 \times 3 \times 0.2$ mms.) with planes (100), (110), and (111) approximately parallel to the largest cross section have been orientated so that the [100], [110] and [111] axes are parallel to the X-ray beam and the (010), (110) and ($\bar{1}\bar{1}$ 0) planes are parallel to the axis of rotation respectively. The diffuse domains studied have been selected close to the origin and include (hh0) and (h00) reflections. Sections of diffuse domains which have been examined are approximately parallel to the (110) and (010) planes. This analysis has been carried out over several weeks since exposure times for the weak diffuse reflections are of the order of days when monochromatic radiation is used. From these preliminary data the shapes of the diffuse clouds around the reciprocal lattice points have been deduced to be ellipsoidal and compare favorably with those calculated from

the elastic constants of germanium by Jahn's formula. The germanium crystal with the (110) plane parallel to its surface has been bombarded with 9 Mev deuterons for 5 micro-ampere hours at a temperature of about -5°C . The diffuse domains of this crystal were again examined at the very same orientations as they were before bombardment. However, because of the long X-ray exposures required, the crystal must be almost completely non-radioactive (no greater than 3 milliroentgen per day) and hence several half lives of 11 days elapsed before this study was continued. Preliminary evaluation of the data obtained after bombardment indicates that a change did occur in the position of the Bragg reflection from both the (220) and (400) planes. This change of the order of $1/2$ degree in the Bragg angle is not localized but persists with vertical movement of the sample. A change in Bragg angle means a change in spacing, therefore a powder film of the single crystal will be taken. This same crystal will be bombarded again for an additional 5 micro-ampere hours with deuterons in order to discover if there are any further changes. However, further experiments must be completed before this change can be firmly established.

During the period of radioactive decay of the Germanium sample above, an identical series of experiments was performed with a silicon single crystal oriented so the $[110]$ axis was parallel to the x-ray beam and the $[001]$ axis was parallel to the axis of rotation.

After the preliminary pictures had been taken, it was bombarded with 9 Mev deuterons for 5 micro-ampere hours at a temperature of about -25°C . Since the half lives of radioactive products produced by deuteron bombardment of silicon are short only a few days need elapse before the second series of photographs can be taken. This is in progress at present but no change has been observed so far. The diffuse domains of silicon have been calculated from the elastic constants and brought to the same relative scale as the germanium values by means of the mean square of atomic vibration in the silicon lattice which was obtained from the Debye temperature. The mean square of atomic vibration of silicon is approximately one half that of germanium, but because of the low absorption of x-rays silicon diffuse spots are more readily recorded than those of germanium.

In order to obtain more extensive data of the diffuse regions in a shorter time, it is proposed to record the diffuse reflections with a Geiger counter mounted on arcs to provide it with two movements, whilst the crystal retains the required third movement. By carrying the Geiger counter on a Weissenberg instrument, instead of the usual film cassette, a desirable, but not necessary, fourth movement can be acquired by means of the equi-inclination rotation. This rotation will allow a greater volume of reciprocal lattice space to be exam-

ined. In this manner it will be possible to scan the diffuse regions by means of sections in three dimensions and hence provide the necessary data for the calculation of the elastic constants.⁽¹⁾

A Geiger counter attachment for the Supper Weissenberg has been obtained, and is now being adjusted and aligned before preliminary experiments are begun. To check the Geiger counter, oxalic acid dehydrate crystals will be investigated; the data obtained will be compared with that reported by K. Lonsdale.⁽²⁾

In order to obtain accurate intensity data a current stabilizer was installed in the present Philips' diffraction unit which will stabilize the emission current to within - 0.1%.

Other phenomena which are sensitive to the degree of regularity of a crystal are Kossel and Kikuchi lines found in X-ray and electron diffraction respectively, observed in germanium and silicon by K. Lark-Horovitz and T.S. Rensema. To examine Kossel lines of crystals before and directly after bombardment by ordinary techniques is rather inconclusive because of radioactive fog.

The presence of Kikuchi lines, however, is not obscured in this manner because of the short exposures and long distances involved. Accordingly a germanium single crystal (N type: 25 ohm cm: 1.5 mms thick) was polished and etched in order to reveal excess and deficiency Kikuchi lines. The crystal, cooled by liquid nitrogen, was bombarded with 9 Mev deuterons for 2 microampere hours. Electron diffraction pictures were taken on both sides of the specimen and compared with those made before bombardment. The same side of the crystal was again bombarded for 3 micro-ampere hours and electron diffraction pictures taken of both sides. No change in the intensity or width of the Kikuchi lines was observed after either bombardment. In this examination, the side on which the deuteron beam was not incident served as an internal standard for all comparisons since the range of the deuterons in the crystal is about 0.2 mm.

Also five germanium single crystals, thicknesses ranging from 0.15 to 1.0 mm, have been bombarded with neutrons at 25°C for one month (fast flux about 5×10^{11} neutrons per cm^2 per sec., slow flux 7×10^{11} neutrons per cm^2 per sec.) X-ray divergent beam, transmission and back-reflection Laue films, and electron diffraction films showed no change in the diffuseness of the Laue spots or of the Kossel and Kikuchi lines. A powdered sample of one of the bombarded crystals gave a cell spacing of $5.656 \pm .001\text{\AA}$ as compared with a measured value of $5.657 \pm .001\text{\AA}$ for an unbombarded sample.

It is however, desirable to make use of the penetrating characteristic of X-rays immediately after irradiation of a crystal in order to investigate its bulk properties and to this end a micro-focus X-ray unit has been constructed and installed.⁽³⁾ The peak specific target loading is about 12 kw./mm.² and thus exposures are a fraction of the present ones and the background due to radioactivity is reduced to a minimum. Preliminary films indicate that in the case of germanium an exposure of 5 minutes with this fine focus tube is equivalent to one of 6 hours with an ordinary tube. Another technique to determine the presence of strains and misorientation of crystallites is to take back reflection powder photographs of single crystals. By this device accurate parameters can be obtained before and after irradiation without grinding the crystal, a process that undoubtedly alters its texture.

An instrument to do this has been designed and constructed similar to the one described by F. W. Mathews and A. G. McIntosh.⁽⁴⁾

Patterns obtained with this device using single crystals are not exact duplicates of normal powder patterns because the apparatus does not give completely random orientations. Therefore, differences in intensities exist between the former and latter patterns. The films obtained show a sufficient number of sharp lines so that if differences occur in lattice parameter, in grain size, or in texture before and after bombardment they should be detected by this method. A selection of silicon and germanium single crystal needles and germanium single crystal plates have been irradiated by neutrons for one month and these will be examined for changes in spacing using this technique. At present only the silicon needles have been studied and these do not show any change in the lattice parameter.

It has been noted in the previous report that radial duplication of Laue reflections of germanium single crystals could be removed by dipping the crystals in liquid nitrogen or by slight heating, but it now appears that such treatment is not necessary as continued exposure to X-rays for about 24 hours has the same effect. Therefore, no exhaustive enquiry of this phenomenon has yet been made as only a few films can be taken with each crystal before this anomaly is removed. Only four out of about thirty germanium crystals have shown duplication and this has been recorded with both filtered and monochromatic radiation from various x-ray tubes. The duplicated reflections are larger than would be expected from the collimating pinholes and the divergence of the beam. When the duplication is removed, the Laue reflections are the size as determined by the collimator. Also the intensities of the single reflections are greater than either

those of the original pairs, and generally it is the inner ones of the pairs which are removed or else coalesce with the outer ones. This duplication exists only over small areas of the crystal (of the order of mm.²) and generally there are a few such areas in one crystal. This fact can possibly account for the reappearance of double reflections after the latter have been removed, as was noted in the previous report. If this is correct then quenching in liquid nitrogen or heating must have little or no effect on the duplicated reflections and the appearance of single ones must be an auto-transformation such as recovery or recrystallization. This change may be accelerated by exposure to X-rays.

No specific reason for this duplication or its removal can be given at present but it seems that such duplication can be obtained by at least four different agents, three of which can be tentatively ruled out because of the information obtained experimentally.

- 1) The presence of some odd type of twinning was considered possible until duplication was shown to persist when the crystal was rotated to random positions about its axis.
- 2) The germanium crystals are mosaic ones with perfect interiors. Duplication can thus arise in Laue spots due to extinction as observed in NaCl and quartz.⁽⁵⁾ However, such duplicated spots can be made single by making the whole crystal mosaic. This fills in the intensity deficient area between the pairs to give single spots which are the same size as the original pairs. In germanium, successive dippings in liquid nitrogen or continuous exposure to X-rays was followed by complete removal of one of the pairs of Laue spots.
- 3) If the diffuse domains of germanium have spikes of intensity as in the case of sodium,⁽⁶⁾ it is conceivable that a few of the Laue spots will be duplicated. However, the diffuse domains of germanium are shown to be ellipsoidal by both elastic constant calculation and by experiment.
- 4) "Radial multiplication of Laue spots may be due to recrystallization about preferential highly strained crystallites".⁽⁷⁾ That is, instead of observing continuous radial asterism due to strain and distortion, discrete spots are observed radially. This discrete spot asterism is observed for Na and K drawn wire at temperatures below room temperature. At room temperature recrystallization takes place and the multiplied Laue spots are changed to single ones. The germanium crystals used have been strained, at least on the surface, as they are ground to the required thickness for X-ray transmission and

also the radially elongated Laue spots points to asterism. On this basis, not only can the duplication be explained but also its removal to give single Laue spots. A more critical examination is called for in order to determine the condition for the production of duplicated Laue reflections and this may be done by grinding germanium with different abrasives.

Germanium needle crystals grown by sublimation (R.E. Davis) also show irregular Laue spot multiplication but this is due to spiral distortion along the needle axis. These crystals are also of interest because they consist of a number of almost regular octahedra embedded in one another along the needle axis. The faces of these octahedra have been recognized as (111) planes by both the optical goniometer and by Laue films. Rotation films show the needle axis to be $[111]$. These octahedra with their well-defined faces allow the needles to be readily orientated in practically any desired manner, this being a prerequisite for the systematic survey of Kossel lines. Silicon single crystal needles supplied by Dupont have also been examined in the very same fashion and the bounding planes have been shown to be (111) and (112) the needle axis being $[111]$. However, there are no characteristically shaped crystallites such as octahedra lying along this axis. Also a silicon oxide, cristobalite is present as powder on these crystals.

Divergent - beam photography^{(8),(9)} produces diagrams geometrically similar to those of Kossel, by placing a crystal in the path of a widely divergent beam of X-rays. In this manner a pattern of reflection and absorption lines in the form of conic sections are recorded and the production of these lines, as noted before, depends on the regularity of the crystal. No widely divergent beam of X-rays is available at present but it is possible to simulate such a beam by means of the Precession camera. In this instrument a collimated beam of X-rays is incident upon a crystal which can be made to precess through any angle up to 30° . This is equivalent to keeping the crystal stationary and precessing the X-ray beam, that is, moving the X-ray beam along the periphery of a cone. Therefore, cone axis films made on the Precession camera with the angle of precession equal to 30° are similar to divergent-beam photographs made with a stationary crystal and a X-ray beam with an angle of divergence of 60° . There is only one qualification, namely, in the case of the Precession camera the narrow parallel beam is the generator of the apparent cones of X-rays. That is, it is a hollow cone and not a solid one as in divergent-beam photography. Therefore, no absorption lines can be recorded and the intense lines appear as arcs of varying intensity due to reflection of both $K\alpha$ and $K\beta$ radiation. The length of these arcs will depend upon the degree of perfection of the crystal. These cone axis films also show displacement and multiplication of Laue cones and arcs due to misalignment of the octahedral crystallites. This work is being continued with a neutron bombardment of germanium and silicon crystals and by indexing the divergent-beam arcs.

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IV. THERMAL CONDUCTIVITY OF GERMANIUM

By J. F. Goff

Results in Temperature Range 65°K to 81°K.

The thermal conductivity for an N-type, antimony doped germanium sample Sb-7 (room temperature resistivity .013 ohm-cm) varies in the temperature interval 65°K to 81°K from about 3.5 watts/cm°K to 2.75 watts/cm°K respectively (Fig. 1). These values are subject to a systematic error amounting to about 1% arising from measurements of the form factor L/A , and to random errors up to 8% attributed to using a vapor pressure thermometer.

Included in the same figure for comparison are results obtained by Estermann and Zimmerman⁽¹⁾ on two ingots cast in graphite crucibles lowered through a vacuum furnace at 3 inches per hour. A "pure" sample with .30 ohm-cm resistivity varies in thermal conductivity from 2.75 to 2.00 watts/cm°K in this temperature range. And an impure sample containing .006 atomic per cent aluminum with resistivity .0021 ohm-cm has a thermal conductivity of about 1.80 watts/cm°K.

Estermann indicates that grain boundaries may play an important part in interpreting his results, implying his specimens may not have been single crystals as was ours.

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Fig. 1—Variation in low temperature thermal conductivity for an N-type single crystal germanium sample.

V. ELECTRICAL MEASUREMENTS ON MOLYBDENUM

By B. W. Randolph

Residual Resistance Studies.

The electrical resistance of 5 mil molybdenum sheets has been measured from room temperature down to 1.5°K . Values in the temperature intervals 77° to 50° , 20° to 10° , and 4.2° to 1.5°K were observed in the usual manner of reducing the pressure over baths of liquid nitrogen, hydrogen, and helium respectively. Residual resistance for the cold rolled samples was reached at about 20°K (Figs. 1 and 2).

Following a three hour anneal at 2400°F in a hydrogen atmosphere, the first sample was cooled at 400°F per hour down to 600°F , when the hydrogen atmosphere was replaced by helium and the cooling continued to room temperature. The surface of this sample was no longer shiny now, but appeared slightly mottled. And the resistance throughout the temperature range had dropped, the residual resistance decreasing from 60 micro-ohms to 48 micro-ohms (Fig. 1).

A second sample was similarly measured (Fig. 2) and annealed for four hours at 2600°F in hydrogen but in the cooling period helium was introduced at about 1000°F . Both surfaces of this sample were discolored by a purple stain, presumably an oxide of molybdenum. This time the resistance throughout the temperature range increased over the cold rolled value, in the residual range rising from 59 micro-ohms to 96 micro-ohms.

By maintaining a hydrogen atmosphere after anneal until the temperature reached 450°F , no surface changes were noted on a third sample. For this sample the residual resistance after annealing was identical to the cold rolled sample. Apparently the annealing process (indicated by recrystallization structure in the X-ray pattern) was not complete.

Since reporting the behavior of the residual resistance of molybdenum sheets at low temperatures before and after deuteron irradiation⁽¹⁾, Mr. Gossick has brought to our attention the work by Thomas and Mendoza⁽²⁾ on the low temperature behavior on molybdenum. These authors found in agreement with earlier experiments by Meissner⁽³⁾, that molybdenum below 15°K seems to go through a minimum and that the resistance increases below 41°K . However, Steiner and Fünfer⁽⁴⁾ have not found such a behavior and their results do not contradict our results.

Fig. 1—Resistance variation with temperature for cold rolled and annealed molybdenum sheet specimens. After annealing surface of sample was slightly mottled.

Fig. 2—Resistance variation with temperature for cold rolled and annealed molybdenum sheet specimens. After annealing, surface of sample was stained purple.

It is interesting that the resistance in our experiments remains constant after annealing since the English group has found that it is even possible that vacuum annealing of molybdenum (obviously not in a perfect vacuum) can introduce compounds which become super-conductors.

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VI. ELECTRON BOMBARDMENT OF GERMANIUM

By E. E. Klontz

Linear Accelerator Program.

The first section of the new linear accelerator producing 4.1 Mev electrons at integrated currents up to one microamp has been put into operation. Passing the beam through an analyzer insures a monoenergetic source, but cuts down the maximum current available to less than 0.1 microamp. A program for utilizing this machine and the completed one having a variable output from 4.1 to 10 Mev has been outlined and a few preliminary tests have been made.

Using an improved cryostat allows bombardments and resistance measurements to be made in vacuum while maintaining temperatures to within a few degrees of liquid nitrogen. An integrating circuit is under construction for making flux measurements.

Preliminary Results.

For testing the behavior of the accelerator, cryostat, and measuring circuits under bombardment conditions, a P-type sample was subjected to a series of bombardments. Initial room temperature and liquid nitrogen resistivities were 1.98 and .22 ohm-cm. respectively.

Three sets of bombardments produced increases in the liquid nitrogen resistivity which ultimately reached the following values for the bombardment times given:

Total Bombardment Time	Resultant Resistivity
2500 sec.	.4 ohm-cm.
9500	21
11500	71

Following each set of bombardments the samples were allowed to return to room temperature; and in each case a corresponding return to very nearly the initial resistivities at both room temperature and liquid nitrogen was noted, characterizing almost complete healing out of defects produced. Further investigations on this sample including a flux curve will follow in the next report.