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SOLID STATE DIVISION

QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING NOVEMBER 10, 1952

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SOLID STATE DIVISION

QUARTERLY PROGRESS REPORT

for Period Ending November 10, 1952

D. S. Billington, Director

Edited by

J. T. Howe

DATE ISSUED

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CONTENTS

	Page
PART I. SECRET PROJECTS	1
SUMMARY	3
RADIATION METALLURGY	6
Creep Under Irradiation	6
MTR Creep Experiment	8
Extensometers	8
Impact Tests on Irradiated Metals	9
High-Temperature Strain Gages	10
ENGINEERING PROPERTIES	12
ORNL Graphite Reactor Liquid-Metals Loop	12
LITR Sodium Stress-Corrosion Loop	12
MTR Fluoride-Fuel Loop	13
FUSED SALTS	15
LITR Irradiation of Reactor Fuel No. 21	15
Liquid Fuels Irradiation in the MTR	15
Dynamic Corrosion Tests	16
Static Corrosion	16
Radiation Effect on the Melting Point of Liquid Fuels	16
Molecular Crystals	18
CRYSTAL PHYSICS	18
Radiation Corrosion of Liquid Fuels in Inconel	18
Proton Bombardment of Aluminum	20
Method of Measuring Dielectric Constants	21
Remote Metallography	22
Effect of LITR Irradiation on Thermocouples	23
High Temperature Thermal Conductivity Measurements on Unirradiated Metals and Alloys	34
NUCLEAR MEASUREMENTS	35
Neutron-Spectra Measurements in the BSF	35
Irradiation Facility for the MTR	36
Nuclear Heating	37

[redacted]

[redacted]

[redacted]

SPECIAL PROJECTS 38

Irradiation of Miscellaneous Materials 38

PART II. UNCLASSIFIED PROJECTS 39

SUMMARY 43

SOLID STATE REACTIONS 44

Low-Temperature Bombardment of Germanium 44
Stable Energy Levels in Bombarded Germanium 44
Magnetic Susceptibility of Germanium 45
Annealing of Potassium Chloride *F* Centers 47
Radiation Effects in Ionic Crystals 48

ENGINEERING PROPERTIES 49

Radiation Stability of Plastics and Elastomers 49

SPECIAL PROJECTS 52

Materials Irradiation 52

[redacted]

[redacted]

Part I

SECRET PROJECTS

SOLID STATE DIVISION QUARTERLY PROGRESS REPORT

SUMMARY

RADIATION METALLURGY

Creep Under Irradiation. The previously reported large creep rates in Inconel have been shown to be a result of erratic behavior of microformer transducers. In the temperature and load range under investigation, postirradiation measurements indicate that the actual deflection is 30 to 40% lower than that indicated by the microformers. Specimens run at 3000 psi and 1500°F in the LITR, on the bench, and in the ORNL graphite reactor show no appreciable difference in creep rate up to 400 hr.

MTR Creep Experiment. The design of a creep apparatus for use in the MTR has been tentatively approved by Phillips Petroleum Co. personnel. Two tubular bellows-loaded tensile creep specimens can be run at the same time, and there will be additional space to accommodate a cantilever creep test and a thermocouple-calibrating device. The apparatus is being assembled for bench tests.

Extensometers. Several coil-spring type transducers were fabricated and tested. The sensitivity is high but there is some inaccuracy resulting from hysteresis. Attempts are being made to achieve a longer and more linear range.

The reproducibility of a Bourdon tube extensometer and its contacts was determined to be 6×10^{-6} to 16×10^{-6} in. over a range of 250×10^{-6} in. A figure-8 Bourdon tube has been designed in an attempt to eliminate the effect of temperature on tube deflection. Present data indicate that the deflection sensitivity increases with temperature, but it is felt that some improvement in the methods used for measuring the deflection must be devised before accurate values can be reported.

Impact Tests on Irradiated Metals. Impact tests have been made on irradiated carbon steel specimens. The transition temperature increases and the fracture energy decreases with increasing irradiation, this is in agreement with the results of tests previously made on ASTM type A-70 steel. Samples of the same carbon steel and type 304 ELC stainless steel have been irradiated in the LITR and will be fractured when the impact tester has been installed in the hot cell.

High-Temperature Strain Gages. A program of tests has been initiated to determine the effects of irradiation on bonded-wire resistance strain gages that are being developed for the HRP by Cornell Aeronautical Laboratory. The gages supplied have been made of Evanohm wire, cemented to and insulated from, a stainless steel plate by either a G-E ceramic cement or a Robert G. Allen Co. ceramic cement. An unbonded wire, suspended in air, was also supplied. Irradiation for one week at 550 to 600°F in hole HB-3 of the LITR caused a resistance increase of 4 to 5%. The change in resistance with temperature is too small to account for the change in resistance attributed to irradiation. Measurements of leakage resistance to ground show that both ceramic cements had high leakage resistances to ground during irradiation.

ENGINEERING PROPERTIES

ORNL Graphite Reactor Liquid-Metals Loop. Another loop was assembled, filled with sodium, and bench tested at 1500°F for about 60 hr, at which time a leak developed in the flowmeter cell at the point where the potential leads were brazed to the cell. Sections of the brazed joint were removed for metallographic examination. The flowmeter was replaced with one having no braze metal in contact with the sodium, and the pump was also replaced with a more efficient pump of the same type. The decay curves of the two sodium loops previously exposed in the graphite reactor were analyzed. No evidence was found of activity other than that of sodium in the sodium contents of either loop.

LITR Sodium Stress-Corrosion Loop. Since the earlier pneumatic-orifice type of extensometer had given difficulties because of seizing and misalignment, a Bourdon-tube type of extensometer was calibrated and attached to the Inconel specimen under test. After the loop was assembled and bench tested at temperatures of 900 to 1100°F for one week, it was installed in hole HB-2 of the LITR and operation was begun. The temperature of operation was 1100°F and has been increased to 1275°F, which will be used for one week before

an attempt is made to reach the intended operating temperature of 1500°F.

MTR Fluoride-Fuel Loop. The design of the loop and shield plug for irradiation of liquid fuels in the MTR is nearly complete. This loop will circulate 4 in.^{3}/min of fuel at 1500°F through an irradiation section that will contain two rectangular specimen plates. All parts of the apparatus in contact with the fuel will be made of Inconel. Fabrication of parts for a prototype pump is 60% complete.

FUSED SALTS

LITR Irradiation of Reactor Fuel No. 21. Reactor fuel No. 21, in an Inconel container, was irradiated at 140 watts/cc in the LITR for 140 hours. Examination shows negligible reaction with the Inconel.

Liquid Fuels Irradiation in the MTR. The thermal flux was determined in the A-38 beryllium piece of the MTR by use of a cobalt wire in an aluminum capsule. The thermal flux was found to be 1.3 $\times 10^{14}$ at a reactor power of 19 megawatts and 1.75 $\times 10^{14}$ at 25 megawatts with the adjacent regulating rod No. 1 in the same position. The extrapolated value for the full power of 30 megawatts was 2.1 $\times 10^{14}$. Three flux capsules were also exposed in the C-48 facility at 50, 83⅓, and 100% reactor power, respectively. Flux data are not yet available.

The reactor power was decreased to 1% of full power and an Inconel capsule containing 0.39 g of reactor fuel No. 30 was inserted in the A-38 facility. Full power was resumed in steps of 3 or 4 megawatts over a period of 24 minutes. The capsule was maintained at 1500°F for 116 hr at full flux. The capsule was shipped to ORNL for chemical analysis and metallographic examination.

Dynamic Corrosion Tests. Design for the dynamic test apparatus for hole HB-2 of the LITR is complete and fabrication has begun. The first in-pile run will be a flux measurement along the length of the capsule to determine the effectiveness of the boral shield.

Static Corrosion Tests. Capsules containing reactor fuel No. 27 were irradiated in the C-48 facility of the LITR for periods of 140 and 565 hours. It is hoped that the two capsules that are now in the facility can be given a 1000-hr exposure or longer. The capsules will be opened for examination when the new remotely controlled metallurgical facilities become available.

Radiation Effect on the Melting Point of Liquid Fuels. After irradiation, when liquid fuel No. 27 cools from the liquid to the solid state, it forms solid crystals in suspension before the remainder of the liquid solidifies. This double melting point of the fuel is being investigated.

The capsule that contains the fuel for which the melting point is to be determined is placed in a well in a solid copper block. A material with a known melting point (that is, sodium chloride) is placed in a second well in a similar location with respect to the surrounding furnace. The temperature of the unknown fuel and the temperature difference between the unknown and the standard are recorded. The equipment has been bench tested with unirradiated fuel and is being prepared for hot cell use.

Molecular Crystals. Work on the unit cell constants of copper phthalocyanine has been completed. The crystal was found to be tetragonal, and the probable space group is $C^1_{4h} - P4/m$.

CRYSTAL PHYSICS

Radiation Corrosion of Liquid Fuels in Inconel. Two fluoride eutectic fuels, Nos. 21 and 27, in Inconel capsules were bombarded with a proton beam of 22-Mev energy. Durations of the bombardments were from 3 to 8 hr for the eutectic No. 21 and from 5.6 to 92 hr for the eutectic No. 27.

A small amount of radiation-induced increase in the intergranular corrosion was observed in the No. 21 fuel capsules subjected to 4-hr or longer bombardments in energy dissipation fields above 3000 watts/cc. No radiation corrosion was detected in the eutectic No. 27 capsules that were bombarded at less than 3000 watts/cc, even for the longer periods of exposure. One capsule bombarded at 3300 watts/cc for 46 hr was found to have intergranular corrosion to a depth of about 0.002 in., as well as a chromium-depleted layer 0.0015 in. deep over the bombarded area. Little increase in chromium content over that of the other bombarded samples could be found by the trace-element analysis. It appears that the corrosion rate at high energy levels rapidly decreases from its initial high value and that total corrosion amounts to only a few thousandths of an inch.

Proton Bombardment of Aluminum. In the investigation into the mechanism of radiation damage to aluminum, two aluminum blocks were subjected to approximately 100 μa-hr of 21-Mev proton bombardments. The blocks were tilted with respect

to the beam so that a range of thicknesses was presented to the beam. After irradiation, hardness measurements were plotted against the distance of penetration. A peak is evident that corresponds approximately to the range for 22.5-Mev protons, this is in good agreement within experimental error.

Experiments are planned to determine whether the hardness change was caused by entrapment of hydrogen atoms or displacement of aluminum atoms.

Method of Measuring Dielectric Constant. A method is proposed for in-pile determinations of the dielectric constant and attenuation factor of insulators in cables. The method is based on the behavior of the input admittance of a terminated quarter-wave line near the quarter-wave frequency the conductance does not vary, and the susceptance changes from positive to negative. An out-of-pile test of RG 11/U cable indicates an accuracy of 0.2% for the dielectric constants and that changes in over-all attenuation of 3% are easily detectable.

Remote Metallography. Design and fabrication of all the major pieces of equipment for remote preparation of metallographic specimens are complete. The equipment is installed in cell No. 2. It consists of a cut-off machine, a mounting press, a Lapmaster, three polishing machines, a specimen washer, an acid-etching machine, and a metallograph. Electrolytic polishing and etching equipment is in process of design and fabrication.

Effect of LITR Irradiation on Thermocouples. An analysis has been made of irradiation effects on the thermocouples used in the thermal conductivity experiments performed in hole HB-3 of the LITR Variation of the thermocouple emf could be correlated with LITR heat power changes caused by neutron-flux-level variations.

High-Temperature Thermal Conductivity Measurements on Unirradiated Metals and Alloys. Apparatus has been fabricated for the out-of-reactor determination of absolute thermal conductivity of metals and alloys. The useful temperature range is from 50 to 850°C.

Irradiation Facility for the MTR. The fast-neutron energy spectrum will be measured in hole HB-3 of the MTR to provide more complete radiation information for the stress-corrosion experiment and other experiments that will utilize this facility.

Apparatus is being designed for inserting, removing, and handling the radioactivants without hazard to the operators and with a minimum delay-time after irradiation. The design will provide a continuous record of the power level within the facility. It will also permit insertion of fission chambers and nuclear-heat measuring equipment.

Nuclear Heating. A calorimeter was used to measure nuclear heat generated in type 347 stainless steel in hole HB-2 of the LITR. Nuclear heat production as a function of position along the axis of hole HB-2 was obtained for a reactor power of 1.5 megawatts. The data are in agreement with previous estimates.

Neutron Spectra Measurements in the Bulk Shielding Facility. Neutron spectra measurements are being made by radioactivation methods at several locations in the Bulk Shielding Facility. A comparison will be made with the results obtained with a proton-recoil counter.

By use of the nuclear reactions $Al^{27}(n,p)Mg^{27}$ and $Al^{27}(n,\alpha)Na^{24}$, the flux at a point 6 in. above the reactor center line in position 27 was determined to be 1.68×10^{11} and 1.23×10^{10} neutrons/cm^2 sec above energies of 4.6 and 8.1 Mev, respectively, at a position 6 in. below the center line, the corresponding fluxes were found to be 1.63×10^{11} and 1.26×10^{10} neutrons/cm^2 sec. The average ratio of neutrons above 4.6 Mev to those above 8.1 Mev was found to be 0.075, compared with a figure of 0.077 for the fission spectrum computed from Los Alamos data. On this basis, the total number of fission neutrons is 2.4×10^{12} neutrons/cm^2 sec. The thermal flux at the two positions averaged 8.1×10^{11} neutrons/cm^2 sec, and the reactor power was 100 kw.

SPECIAL PROJECTS

Irradiation of Miscellaneous Materials. Fast-neutron-flux bombardment of diamond and cubic silicon carbide in the C-39 position in the LITR for a period of nine weeks has caused a continuing lattice expansion.

Irradiation of powdered alpha quartz under the same conditions produced a lattice expansion comparable to that in diamond. Single-crystal studies will be undertaken.

Materials Irradiation. Materials under irradiation at Hanford and those being prepared for irradiation include two types of tool steels and ceramics samples.

Preirradiation measurements of modulus of elasticity, electrical resistivity, density, and hardness have been made on specimens of thorium metal.

No significant changes were found in antiferromagnetism of Fe_2O_3 powder that had been irradiated in hole 51 of the ORNL graphite reactor.

RADIATION METALLURGY

CREEP UNDER IRRADIATION

W. W. Davis J. C. Wilson
J. C. Zukas

During this quarter, tentative results from cantilever creep tests on Inconel at 1500°F in both the ORNL graphite reactor and the LITR showed that, compared to bench tests, there was less creep under irradiation at the 1500-psi stress level and about the same amount of creep under irradiation at the 3000-psi stress level. Postirradiation measurements of the total creep on two previously reported tests,[1,2] in which abnormally large creep strains were observed during irradiation, indicate that these values were high because of erratic behavior of the microformer transducers used to measure the deformation. Several tests this quarter were negated by microformer failure.

At 1500°F and 1500 psi, the curves in Fig. 1 for both the tests in the LITR and the ORNL graphite reactor fell below that of the latest bench test and generally below the average of three previously reported bench tests.[3] During the in-pile test of an earlier specimen under the same conditions of stress and temperature, the deformation,[1] as indicated by the microformer during irradiation, was 0.108 in., but postirradiation measurements showed that the deflection was only 0.036 inch. The latter figure is approximately twice that observed in bench tests but less than one-third of the recorded in-pile deflection, consequently, the possibility still exists that this specimen, annealed at 1650°F rather than at 1700°F as were the later specimens, may have exhibited abnormal behavior under irradiation.

Similar postirradiation measurements on a test performed at 1500°F and 2000 psi again showed that the actual deflection was 40% less than that indicated by the microformer during irradiation.[2] At the end of the irradiation, the in-pile test was operated for several hundred hours at 1600°F. No corresponding bench data were procured, so the total deflection cannot be compared with bench test data.

The measurement of the creep deflection in the irradiated specimens was carried out on an existing

[1] W. W. Davis, J. C. Wilson, and J. C. Zukas, *Solid State Quar Prog Rep Aug 10, 1952*, Fig. 1, Curve *a*, ORNL-1359 (in press).

[2] *Ibid*, Fig. 2, Curve *a*

[3] *Ibid*, Fig. 1, Curves *C*, *D*, and *E*

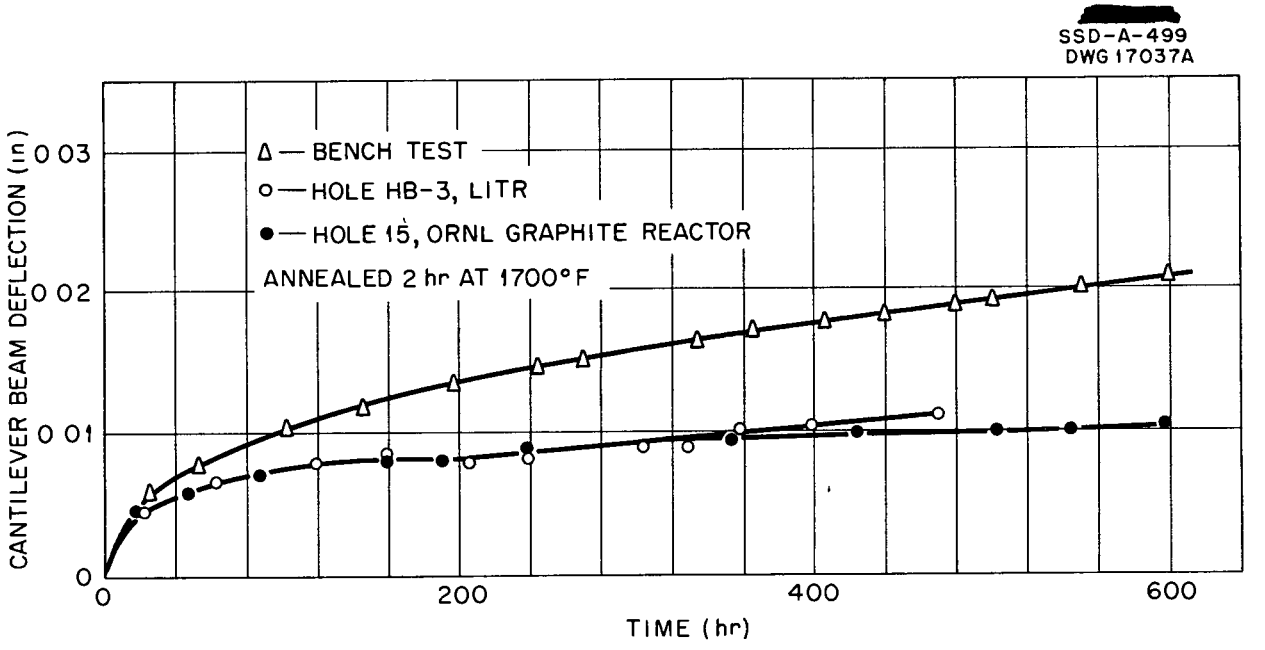

Fig. 1. Cantilever Creep Tests on Inconel at 1500°F and 1500 psi.

remotely controlled profilometer. A movable table on the machine traverses the top of the specimen[4] under a gage head, and the position of the gage head and the table are simultaneously recorded in rectangular coordinates on a strip-chart recorder. Measurement of the angle between the chart record for the top of both the stationary end of the specimen and the loading beam was found to bear a simple relationship to the creep deflection for a large number of bench-test specimens used for calibration. All available irradiated in-pile specimens are now being measured.

Figure 2 shows that at 3000 psi and 1500°F there is no substantial difference up to 400 hr between creep tests run in the LITR, on the bench, and in the ORNL graphite reactor. The curve for the latter test is concave upwards after 350 hr, however, because faulty operation of the temperature controller permitted only intermittent operation at 1525°F after 350 hr, no particular significance is attached to this behavior. In the interval between 200 and 350 hr, the creep rates for all three curves are virtually the same.

[4] D. S. Billington, *ANP Quar Prog Rep Dec 10, 1950*, ORNL-919, Fig. 9 3, p. 228.

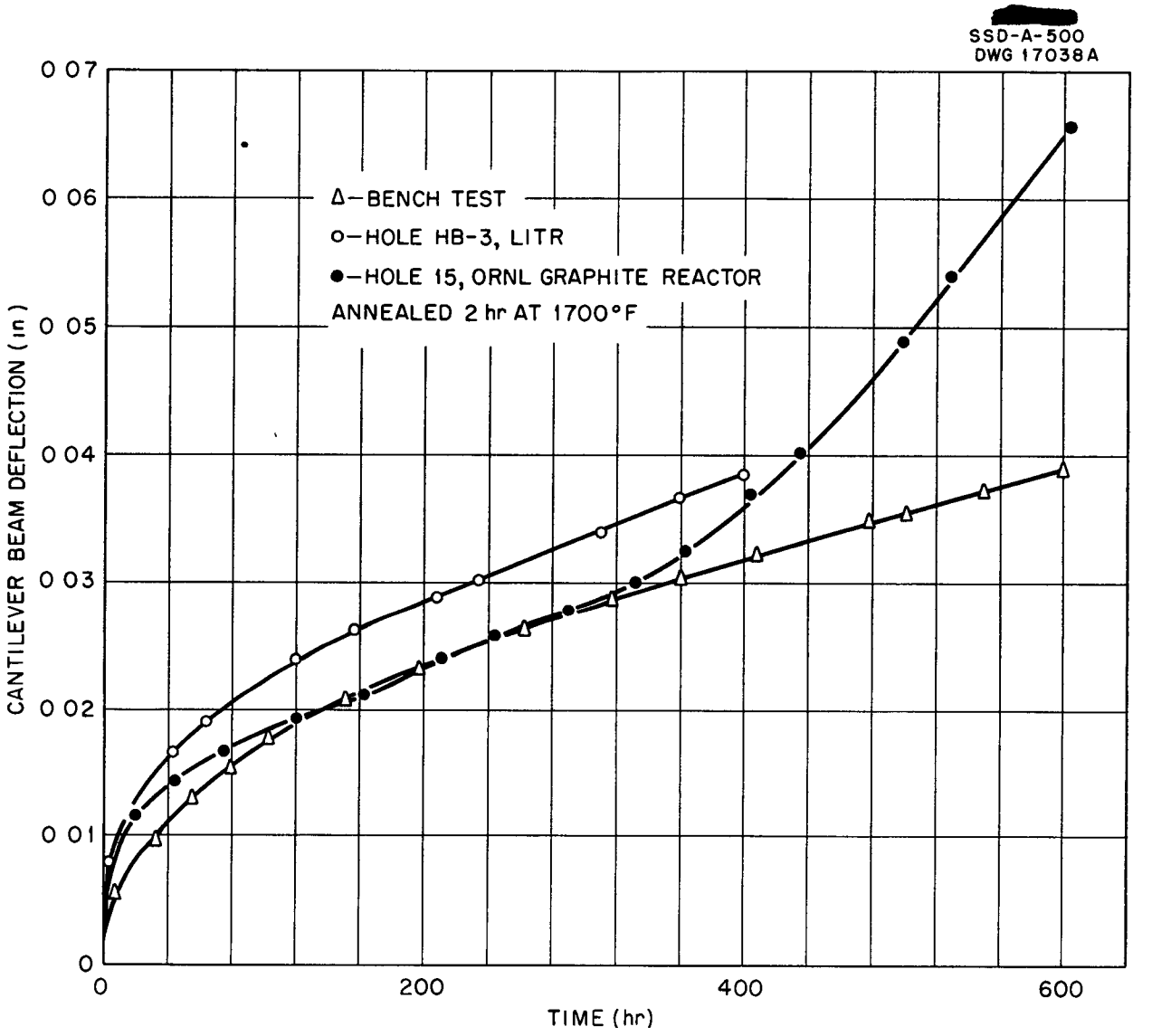

Fig. 2. Cantilever Creep Tests on Inconel at 1500°F and 3000 psi.

MTR CREEP EXPERIMENT

J. C. Wilson

A design for a creep apparatus for the MTR has been sent to Idaho Falls, and notice has been received that the design is acceptable, pending further study. The design contains two tubular bellows-loaded tensile creep specimens, and it is expected that a cantilever creep test and a thermocouple-calibrating device will be added later. Most of the component parts have been fabricated and partly assembled for test. Further assembly will proceed after the calibration of the bellows and the completion of a few minor design changes. Calibration is required to determine the effective area and spring rate of the bellows so that the stress on the specimen may be computed, and a device for performing the calibration has been constructed. The device is composed of an enclosed chamber that communicates with the bellows and contains a proving ring that carries SR-4 strain gages for external electrical indication of the force exerted by the bellows under test pressures.

Because the constancy of stress in the bellows-loaded apparatus is dependent on the behavior of the gas-pressure controlling valve, a comprehensive series of tests on several commercial makes of valves was run by W. R. Miller of the Instrument Department. The relative constancy, drift, return to zero, and controllability of downstream pressure where downstream pressure buildups occur, and several other properties were tested. The Moore model 40-30 and the Kendall model 10-30 proved superior to the five other makes of values tested, the Kendall has the additional advantage of consuming no gas on dead-end service and consequently is more economical if inert gases are used in the system.

A suggested design for the safety circuit was completed by J. T. DeLorenzo of the Instrument Department, but late receipt of the full MTR specifications for safety circuits has necessitated some changes before a proposal can be submitted to the Phillips Petroleum Co. Provisions were made for connection to either the LITR or the MTR safety circuits so that the complete system may be tested in operation in the LITR before shipment. All components for this system have been received, but delivery of the temperature controllers has been rescheduled to a later date for the third time. Several power panels and thermocouple patch boards for the furnace system have been designed and built.

EXTENSOMETERS

J. C. Wilson R. A. Weeks

Several coil-spring-type transducers[5] were fabricated and tested. Each consists of a tightly wound single-layer helical coil of 0.0056-in.-dia gold-plated Monel wire. The feed angle of the wire was varied continuously during winding so that the adjacent turns tend to separate serially upon tensile deformation. The coils tested were wound to a length of about 0.15 in. on an 0.05-in. round mandrel. The sensitivity is high, for an extension of 0.005 in., the resistance of the coil changes from 0.8 to 1.4 ohms. However, hysteresis was found to be as great as 0.001 inch. The hysteresis was reduced at least 60% by applying vibration to the system by means of a vibrating marking stylus held lightly against the micrometer head on which the coil was mounted. A longer helix is being fabricated in an attempt to achieve a longer and more linear range.

The make-break sensitivity of the razor blade contacts used with the Bourdon-tube extensometer[6] was determined to be at least 5×10^{-6} inch. Vibration prevented these measurements from being made on the Templin calibrator, therefore elastic strains introduced in a tensile specimen by loading in a testing machine were utilized to move the contacts over known increments in the microinch range for this test. Closure of the contacts was indicated by a thyratron relay circuit, the contacts are in the grid circuit of a thyratron tube, and negligible arcing occurs on closure. After the make-break sensitivity had been determined, the reproducibility of readings of a Bourdon tube–contact system was determined by measuring the pressure required to distend the tube to remake contact after the razor blades had been separated in known increments of about 125×10^{-6} inch. The reproducibility was shown to be in the range of 6×10^{-6} to 16×10^{-6} in. for a series of four trials involving both upward and downward motion of the contacts over a range of 250×10^{-6} inch. Pressure measurements with a precision of approximately 0.25% were possible with a Baldwin type EMB load cell in conjunction with a Baldwin-Foxboro portable strain indicator.

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(5) W. A. Wildhack and T. A. Perls, *Quarterly Progress Report of the Office of Basic Instrumentation for the Quarter Ending December 31, 1951*, NBS-1467, p. 19.

(6) J. C. Wilson and R. A. Weeks, *Solid State Quar Prog Rep May 10, 1952*, ORNL-1301, Fig. 4, p. 9.

The figure-8 geometry shown in Fig. 3 was chosen in order to study the effect of temperature on Bourdon tube deflection, because thermal expansion should cause no change in position of the free end of the tube relative to the mounting point. In addition, it was reasoned that the radial components of distention of each branch of the tube should cancel, and the resultant motion of the free tip should then be linear (in a direction tangent to both circular branches of the tube at their point of common tangency) rather than arcuate. The deflection sensitivity of such a tube is about 0.0005 in./psi of applied pressure. The deflection was measured between 0 and 80 psi for temperatures up to 370°F. The data obtained thus far show that the deflection sensitivity increases with temperature (approximately 0.05%/°F at constant pressure), but a more refined method of measuring this quantity must be devised before accurate values can be reported.

IMPACT TESTS ON IRRADIATED METALS

R. G. Berggren N. E. Hinkle

Impact tests on a normalized carbon steel (described previously[7] as SAE 1040 steel, but found by analysis to be of the composition: 0.167% C, 0.68% Mn, 0.208% S, 0.12% P, 0.04% Al) were made and the data plotted (Fig. 4) for unirradiated control specimens and specimens irradiated for periods

[7] R. G. Berggren and R. H. Kernohan, *Solid State Quar. Prog. Rep. Jan. 31, 1952*, ORNL-1261, p. 20.

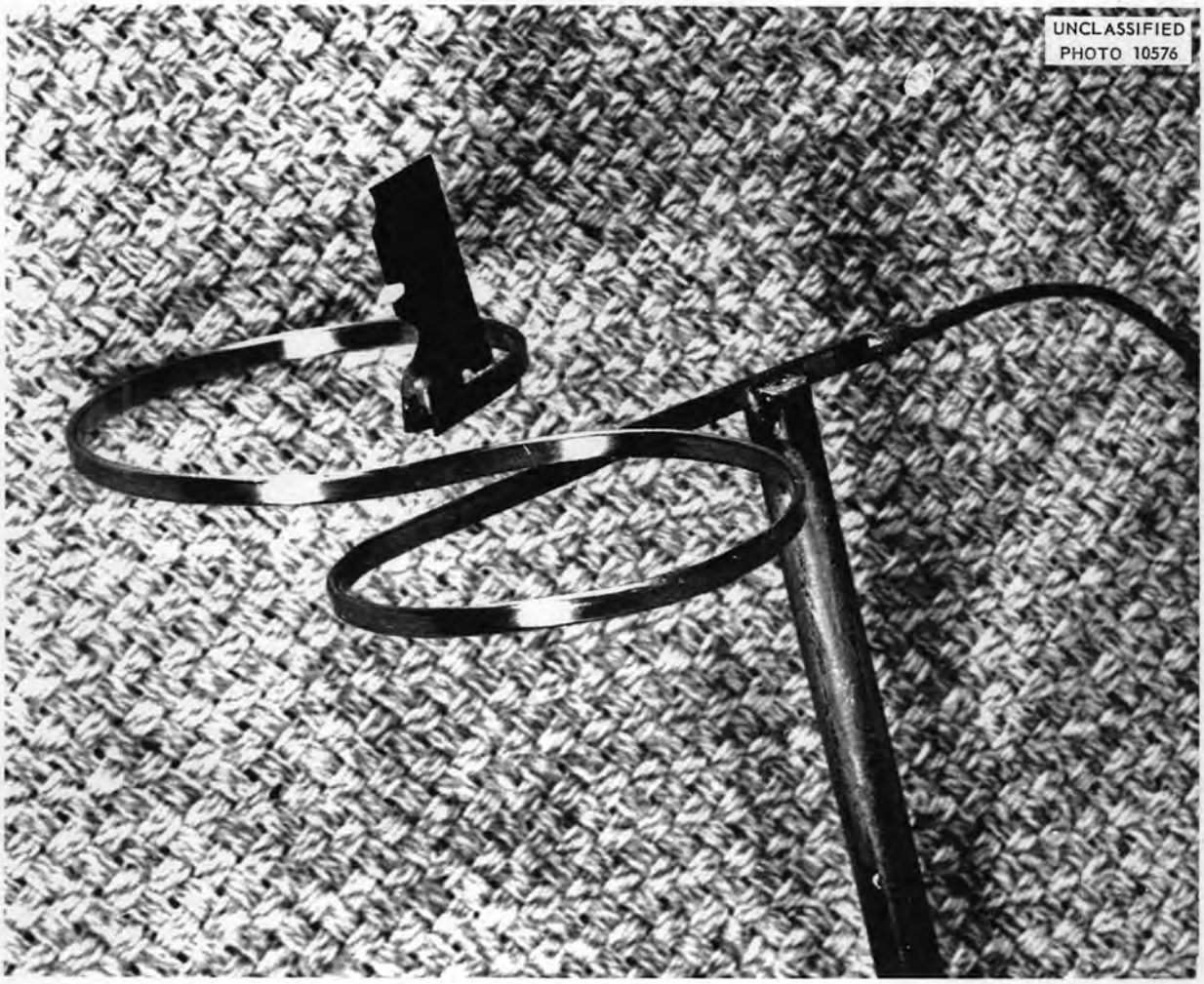

Fig. 3. Figure-8 Bourdon Tube.

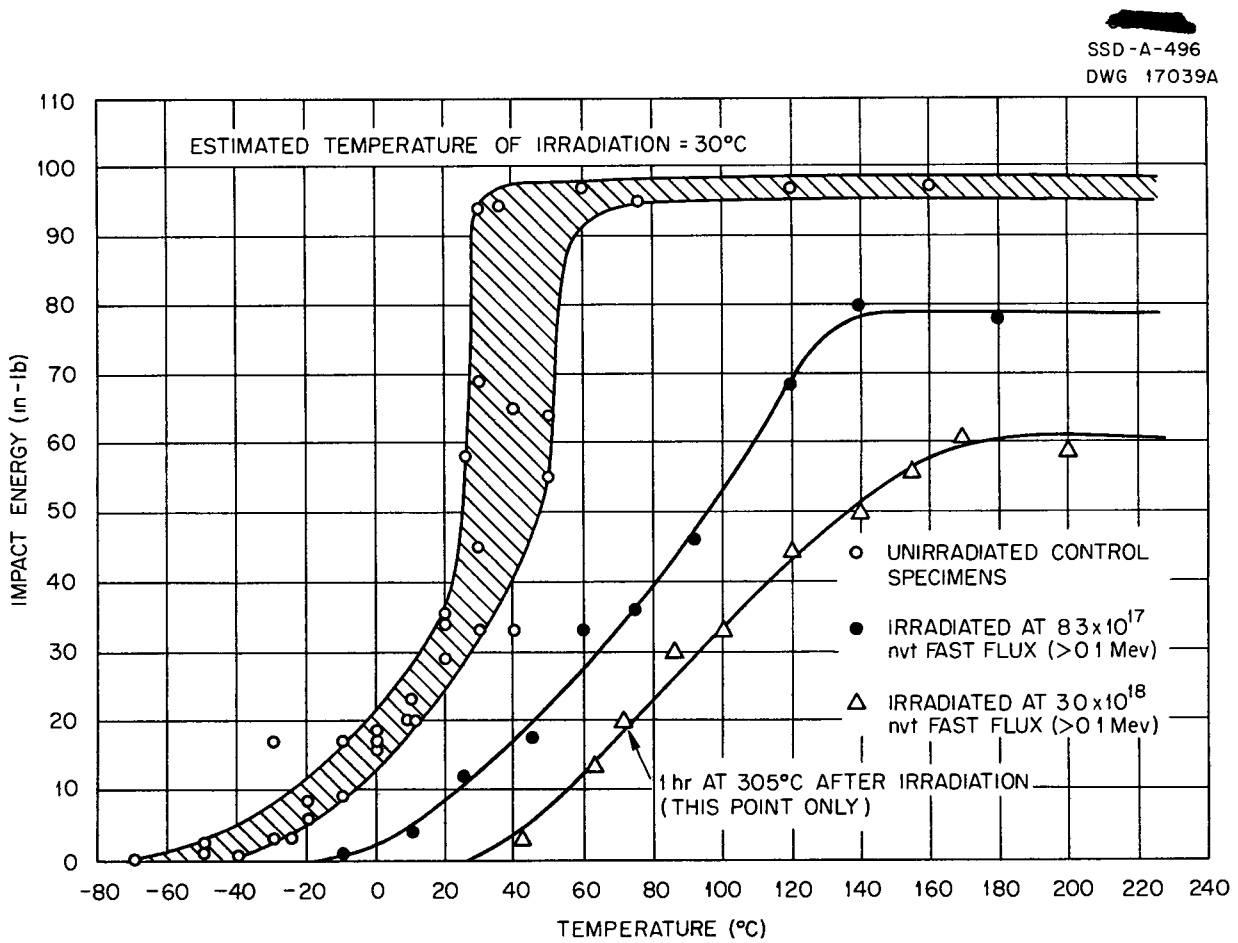

Fig. 4. Impact Strength of Irradiated Carbon Steel.

of four and ten weeks (integrated flux above 0.1 Mev equal to 8.3×10^{17} nvt and 3.0×10^{18} nvt, respectively) in donut hole 1867 of the ORNL graphite reactor. The curves show that the transition temperature increases and the fracture energy decreases with increasing irradiation. This is in accord with the results of tests on ASTM type A-70 steel reported previously.[8] One fracture test was made on a specimen that was heated for 1 hr to 305°C after irradiation in an attempt to anneal out the damage. The impact strength agrees with that of similar specimens that were only irradiated.

Samples of type 304 ELC stainless steel and the steel described above, irradiated in C-59 (vertical beryllium exposure piece) of the LITR, will be fractured when the impact tester has been installed in the hot cell. A new coolant system and a hydraulic sample positioner are being fabricated for the impact tester.

Impact tests of unirradiated iodide and commercial titanium have been made in collaboration with the Metallurgy Division and are reported in the HRP quarterly report.[9]

HIGH-TEMPERATURE STRAIN GAGES

R. G. Berggren J. C. Wilson

An irradiation program has been initiated to test the performance of the bonded-wire resistance strain gages that are being developed for the HRP by Cornell Aeronautical Laboratory (CAL).

Preliminary tests have been made on two types of bonded gages supplied by CAL, as well as on an unbonded-wire element. Each gage consists of

[8] R. G. Berggren, *Solid State Quar Prog Rep. Aug 10, 1952*, ORNL-1359 (in press).

[9] E. C. Miller *et al*, *HRP Quar Prog Rep Oct 1, 1952*, ORNL-1424, p. 120.

a length of 0.001-in.-dia Evanohm[10] wire. The gages supplied by CAL have the wire cemented to, and insulated from, a type 310 stainless steel plate by either a G-E ceramic cement or a Robert G. Allen Co. ceramic cement. The unbonded wire was suspended in air by the lead wires.

One of each of the gages was irradiated in an unstrained condition for a period of one week in hole HB-3 of the LITR at a temperature of 550 to 600°F. The resistance of the gage element and leakage resistance to ground were measured while the gages were in the reactor. The data obtained are plotted in Fig. 5 and show that the resistance of all three gages increased 4 to 5% during irradiation. The temperature coefficient of resistance between 571 and 610°F was determined for the unbonded wire after a 92-hr exposure and for the wire gage bonded with the Allen cement after a 141-hr exposure. Both coefficients were negative over the temperature range measured, whereas an unirradiated length of wire from the same spool gave a positive coefficient over the same temperature range. Since the points in Fig. 5 at 138 and 158 hr, taken at 505 and 415°F, respectively, lie above the smoothed curve that was measured in the range of 550 to 600°F, they furnish additional evidence that the temperature coefficient of resistivity was negative. The change in resistance with temperature is an order of magnitude too small to account for the change in resistance attributed to irradiation.

No data on the metallurgy of the Evanohm alloy are available, but there is some evidence[11] of an ordered structure in nickel-chromium alloys of approximately the same composition as Evanohm (without the Cu and Al), and the sign of temperature coefficient of resistance varies with temperature.

Measurements of resistances between the gage wires and ground show that both ceramic cements had high leakage resistance during irradiation.

[10] Typical analysis 74.50% Ni, 20 00% Cr, 2 75% Cu and 2 57% Al manufactured by the Wilbur B. Driver Co.

[11] Z. Yano, *Japan Nickel Rev* 9, 17 (1941).

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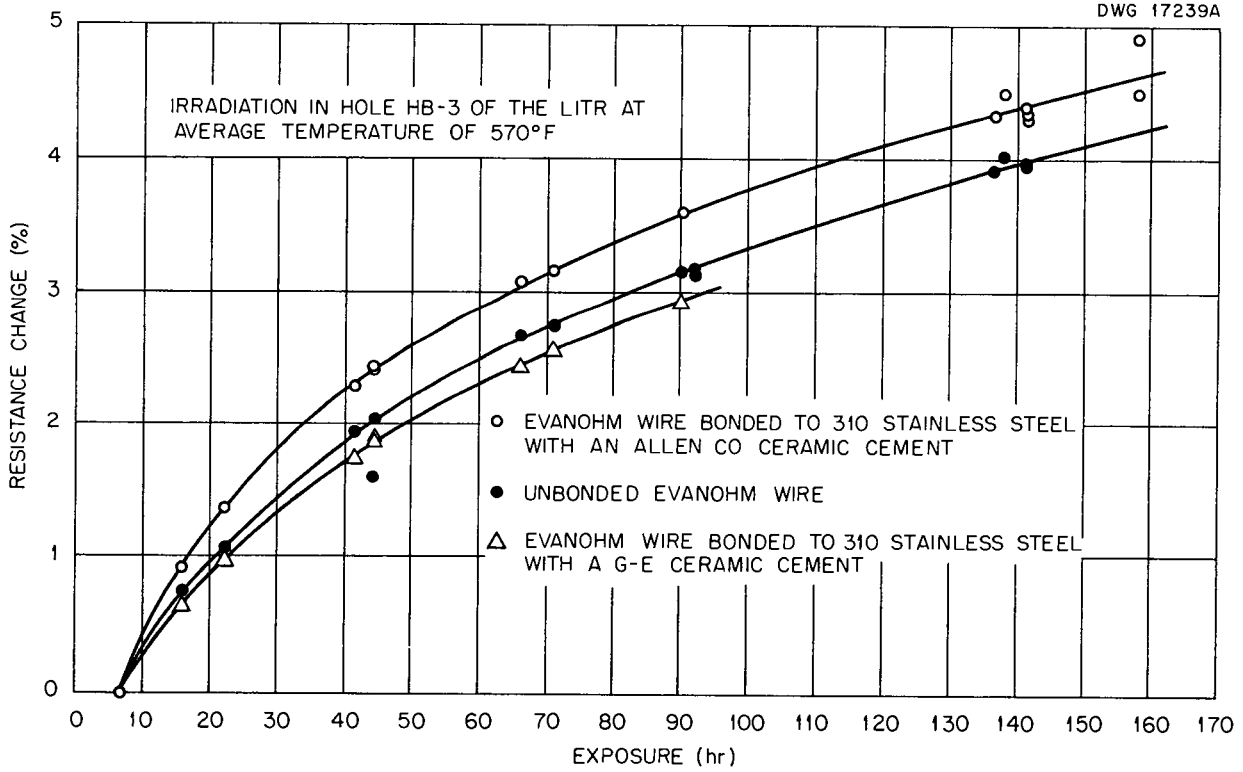


Fig. 5. Effect of Neutron Bombardment on Resistance of Cornell Aeronautical Laboratory High-Temperature Strain Gages.

The gage bonded with Allen cement showed a resistance to ground of 8.5 megohms and higher during irradiation and 580 megohms after completion of irradiation. The gage bonded with G-E cement had a resistance to ground of 40 megohms and higher during irradiation, the resistance to ground after completion of irradiation was not measured because the gage failed after 90 hr of irradiation for reasons not known. The resistance to ground of the unbonded wire was 18 megohms and higher during irradiation and 480 megohms after completion of irradiation.

ENGINEERING PROPERTIES

ORNL GRAPHITE REACTOR LIQUID-METALS LOOP

C. D. Baumann, C. Ellis, F. M. Blacksher, M. T. Morgan, R. M. Carroll, O. Sisman, W. W. Parkinson

A continuing program of investigation into the compatibility of high-temperature coolants and structural materials under reactor irradiation is being carried out in which Inconel systems containing flowing sodium are exposed in the ORNL graphite reactor. These systems, described in a previous quarterly report[1] as consisting of a loop of Inconel tubing connected to a surge tank and an electromagnetic pump cell and flowmeter, have been revised to include a bypass circuit that contains a cooling tank and a stainless steel micrometallic filter to remove sodium oxide from the stream. During bench tests, the two previous loop systems had developed leaks at the in-pile end block. Another loop was assembled and filled with sodium. After a bench-test of about 60 hr at 1500°F, a leak developed in the flowmeter cell where the potential leads were brazed to the cell with nickel-palladium brazing alloy. Since there were indications that the sodium had attacked the brazed joint, sections for metallographic examination were cut from the affected joint and from joints exposed to sodium at lower temperatures.

The loop was rebuilt and included a new flowmeter on which the potential leads were terminated on the outside of the cell instead of by the method described in a previous report.[2] This eliminated the contact of sodium with nickel-palladium alloy. Also, a new pump cell was installed to eliminate the nickel-palladium joints previously used, layers of silver sheet were substituted for nickel sheets in the laminated current leads This should reduce the resistive power loss in the pump and improve its efficiency. The loop assembly has been leak tested, and heaters are being attached before it is filled with the sodium charge.

The decay curves of the two sodium loops that had been exposed in the graphite reactor[3] were analyzed by utilizing the calibration of the ionization chambers described last quarter.[1] No evidence of activities other than that of sodium was found in the sodium contents of either loop

A topical report on the lithium loop[4] and its bremsstrahlung activity, for which the chambers above were calibrated, is in preparation.

LITR SODIUM STRESS-CORROSION LOOP

W. E. Brundage, C. Ellis, F M. Blacksher, A. S. Olson, R. M. Carroll, W. W. Parkinson, O. Sisman

Ir order to study liquid metal systems at fluxes higher than that of the ORNL graphite reactor, an Inconel loop has been under construction for insertion in the LITR. After fabrication of the parts for the loop and the shield plug upon which it was mounted, the high-temperature calibration of the extensometer to measure creep of the Inconel specimen was carried out.

Because the earlier pneumatic-orifice type of extensometer had given difficulties that resulted

(1) C. D Baumann *et al*, *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

(2) C D. Baumann *et al*, *Solid State Quar Prog Rep Jan 31, 1952*, ORNL-1261, p. 21, C. D. Baumann *et al.*, *Solid State Quar Prog Rep May 10, 1952*, ORNL-1301, p. 11.

(3) R. M. Carroll, *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

(4) C. D. Baumann, R. M. Carroll, and O. Sisman, *Physics of Solids Institute Quar Prog Rep April 30, 1951*, ORNL-1095, p. 69, C D. Baumann, R. M. Carroll, O. Sisman, W. W. Parkinson, and C. Ellis, *Physics of Solids Institute Quar Prog Rep July 31, 1951*, ORNL-1128, p. 35

from seizing and misalignment, a new extensometer was made that utilized a Bourdon tube.[5] A knife-edge electrical contact, brazed to the movable end of the Bourdon tube, opposed a similar contact attached to one end of the specimen. In this manner, the position of the movable end of the Bourdon tube is located with respect to the fixed end, which is attached to the other end of the specimen. The Bourdon tube was made of ⅛-in.-OD Inconel tubing that was electrostripped to a wall thickness of 0.005 in., rolled flat, and bent into an S shape to conserve space. The extension of the tube could be determined by measurement of the pressure applied to it. In order to find the relation between extension and pressure at various temperatures, an inert-atmosphere furnace for the extensometer was constructed and fitted with a suitable movable contact attached to a dial gage that was sensitive to 0.0001 inch. The calibration was carried out at 70, 877, and 1508°F. At the higher temperature, the Bourdon tube was found to be subject to fairly rapid creep so that a standard rate of application of pressure had to be adopted. At a rate of 1 lb/sec, all the creep was recoverable within 10 min for extensions up to 0 015 in., the pressure required for this extension was about 60 psi. The extension at each temperature, when the standardized rate of pressure application was followed, was directly proportional to the pressure, although for different temperatures the extension varied for a given pressure. The calibration curves were found to be reproducible to 0 0007 inch.

The calibrated extensometer was attached to the specimen, and the various parts of the loop were assembled on the shield plug. The loop was given the usual leak tests, fitted with heaters and thermocouples, and filled with sodium. In order to allow access to the loop after bench testing, the stainless steel tube sections, which enclosed the loop in a helium atmosphere and provided an outer vacuum jacket, were fixed in place but not welded.

The loop was bench tested at temperatures of 900 to 1100°F for one week in order to accomplish three objectives (1) to check the entire system for leaks when it was exposed to sodium at high temperatures, (2) to free the sodium in the loop of oxide by means of the bypass circuit which contained a cooling tank and a filter, and (3) to determine whether a temperature of 1500°F and a flow of 1 ft/sec could be attained in the creep-specimen portion of the loop.

During the bench test, the outer plug, which carried a water-cooled jacket, was inserted in hole HB-2 of the LITR. An instrument panel and a gas and vacuum control panel were also completed and moved into position beside the LITR. After successful completion of the bench test, the tube sections that formed the helium enclosure and the vacuum jacket were welded in place and leak tested. The bypass circuit was cut off the loop, and the connecting tubes were closed in final preparation for in-pile operation.

On October 21, the loop and the shield plug were inserted in hole HB-2 of the LITR. A lead-brick shield was erected around the electromagnetic pump located outside the pile, and the circulation of sodium was started. The loop was operated at a temperature of about 1100°F rather than at the intended operating temperature of 1500°F because of the recent difficulty with nickel-palladium braze[6] and the discouraging results of current tests of this braze by the Metallurgy Division. After 16 hr of operation, one of the leads of the cylindrical heater around the creep specimen opened. This end of the heater also grounded, so that, by use of an isolating transformer, 220 volts could still be applied to the heater. However, after several hours, the remaining lead failed. By increasing the power to the other heaters, operation was continued and the temperature was gradually increased to 1275°F. In order to conserve the life of the remaining heaters, it was decided to operate for one week at this temperature before attempting to reach 1500°F.

MTR FLUORIDE-FUEL LOOP

W. W. Parkinson O. Sisman

An experiment is being devised to study the dynamic corrosion of Inconel by fluoride fuels under irradiation in the MTR.[1] The design of the loop and the shield plug upon which it is mounted for insertion in a beam hole of the MTR is virtually finished. The work remaining to be done on the design consists of checking some of the drawings for errors. About 90% of the materials for

[5] C. D. Baumann *et al*, *Solid State Quar Prog Rep May 10, 1952*, ORNL-1301, p. 11.

[6] See "ORNL Graphite Reactor Liquid-Metals Loop," this report.

the loop and its associated apparatus has been ordered and about 50% has been received.

A schematic drawing of the apparatus is shown in Fig. 6. A shield plug for insertion into a reactor beam hole will fit inside three jackets that will enclose the loop mounted on the end of the plug. Cooling water will circulate between the outer two jackets, and the space between the middle and inside jackets can be evacuated or filled with inert gas. The innermost jacket will enclose the loop in an atmosphere of helium, and all three jackets will be made of stainless steel for safety in event of a fuel leak. The jackets and plug are designed for easy removal of the inside plug and loop in order to provide access to the loop and associated equipment.

The air motor to drive the fuel-circulating pump will be supported at the end of the plug on concentric air lines. The loop will consist of a venturi flowmeter, a surge tank, a U-shaped irradiation section, and a heat exchanger. The air-driven centrifugal pump will force the fuel first through the venturi. The pressure taps of the flowmeter will be connected by small tubes to liquid-separator tanks that will allow the transmission of the fuel pressures at the venturi through inert gas to pressure cells for indication of flow. The fuel will flow from the venturi into the surge tank that serves to remove bubbles from the stream and to provide a reservoir for thermal expansion. The discharge end of the surge tank will have a tapered transition section to avoid turbulence and entrainment of bubbles. The fuel will flow from the surge tank through the U-shaped irradiation section that will contain two rectangular specimen plates at points nearest the reactor lattice. The U section will be made of small tubing in order to minimize the volume of fuel irradiated and the fission-heat generation. A compromise in the size of this tubing is necessary, however, because the velocity of flow must be in the turbulent region for satisfactory removal of heat, and the diameter of the tubing must be large enough to maintain the pressure drop at this velocity within the operating range of the pump.

A cylindrical heat sink, along the sides of which will lie the U-shaped irradiated tubing, will be a special safety feature of the irradiation section.

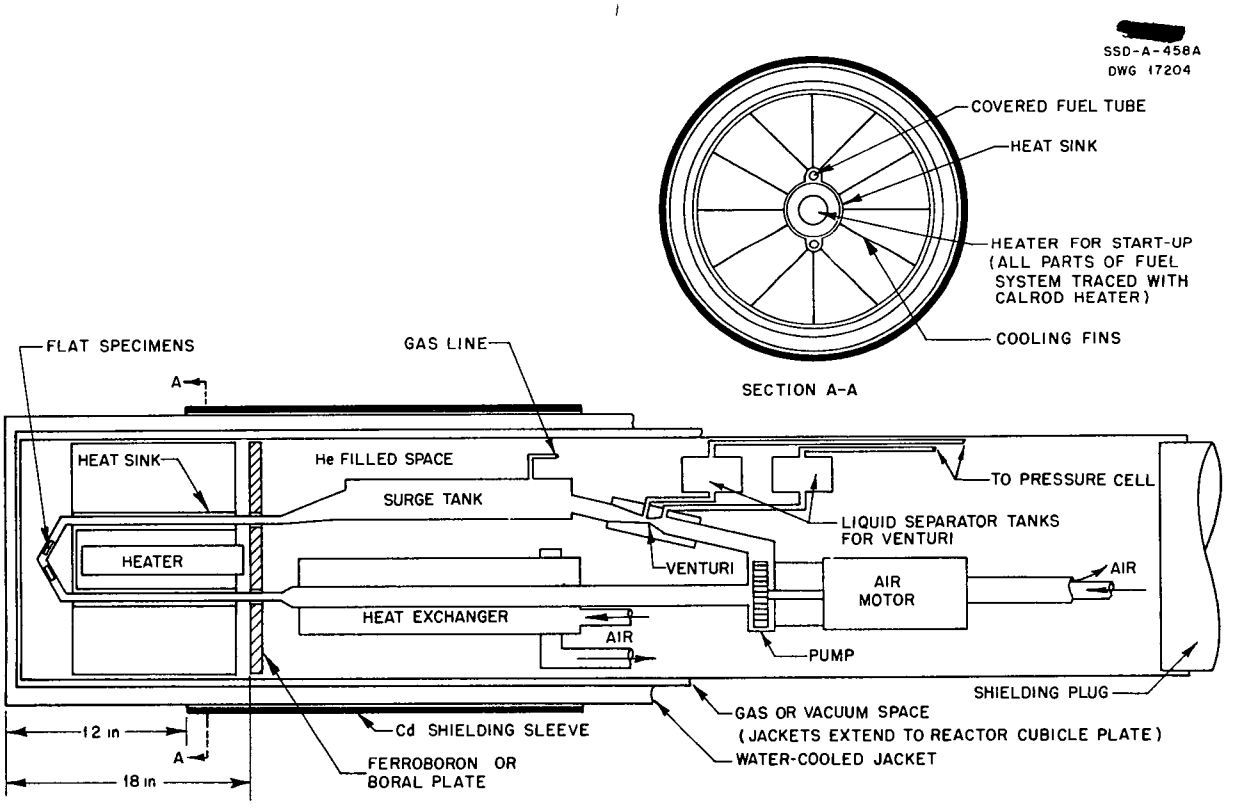


Fig. 6. Dynamic Corrosion Apparatus for MTR Experiment 2A.

The tubing will be covered with massive strips of metal brazed to the main mass of the heat sink. Longitudinal fins to conduct and radiate heat to the walls of the inner jacket and thence to the cooling water will radiate from the heat sink. The function of the heat sink will be to avoid a rapid temperature rise in the fuel and its containing tube in the event that a flow stoppage prevents the normal heat removal by the circulation of fuel.

After leaving the irradiation section, the fuel will pass through the air-cooled heat exchanger where sufficient fission heat will be removed to maintain the temperature of the loop at about 1500°F. From the heat exchanger, the fuel returns to the inlet of the pump for recirculation. The design values are temperature, 1500°F, flow, about 4 in.^{3}/min, tubing for the irradiation section, 0.175 in. ID, pressure drop, about 26 psi. The density of heat generation will reach about 2000 watts/cc, and the heat exchanger is designed to remove about 7500 watts.

In order to reduce the fission heating of the large volumes of fuel in the surge tank and heat exchanger, it was thought necessary to reduce the neutron flux in their vicinity. This will be done by enclosing the jackets around these parts in a layer of cadmium and by inserting a ferro-boron plate between the irradiated section and the rest of the apparatus.

All parts of the apparatus in contact with the fluoride will be made of Inconel. Construction is under way on only the pump and its drive motor. The fabrication of parts for a prototype pump is 60% complete, and a commerical air motor is being modified so that it will join directly to the pump housing.

FUSED SALTS

LITR IRRADIATION OF REACTOR FUEL NO. 21

G. W. Keilholtz M. J. Feldman

The reactor fuel No 21[1] was irradiated at 140 watts/cc in the LITR for 140 hr at a flux of 2.3×10^{13} Metallurgical and chemical examinations show no appreciable reaction of the fuel with the walls of the Inconel container that can be attributed to radiation effects. There was less than 1 mil penetration of the liquid into the Inconel in 140 hours

LIQUID FUELS IRRADIATION IN THE MTR

J. G Morgan H E. Robertson
A. E Richt P R. Klein

Installation of the ORNL-2b fused-salt testing facility was completed at the MTR site The guide tube extended from a $1\frac{3}{8}$-in. hole in a beryllium "A" piece at position A-38 through a specially designed flange in the north tank access hole. The control and recording instruments were placed on the north balcony. In order to maintain the capsule temperature at 1500°F, controlled cooling air is forced past the capsule and discharged into hole VG-25. The salt is contained in an Inconel capsule, 0 200 OD × 0 100 ID, and an active column of 1 inch. The capsule, with thermocouples attached, is charged down the inner guide tube and seats on a support at the bottom of the tube.[2] The desired capsule temperature is maintained by 7 cfm of air at 45 psi. The contracted air passage around the active end of the capsule permits a cooling air velocity of 430 lin ft/sec.

As a preliminary experiment in the liquid fuels investigation, it was necessary to determine the thermal flux at the capsule position. A cobalt wire in an aluminum capsule was inserted in the experimental tube in the A-38 beryllium block that extends to the center line of the fuel elements. Two such flux measurements were made in order to determine the wattage produced by fission in the test salt One run at a calculated reactor power of 19 megawatts resulted in a thermal flux measurement of $1\ 3 \times 10^{14}$ The other determination at a reactor power of 25 megawatts showed a thermal flux of 1.75×10^{14} The extrapolated value of flux at full power, or 30 megawatts, was $2\ 1 \times 10^{14}$. The position in A-38 is adjacent to regulating rod No 1, and thus the flux is likely to be very sensitive to variations in the rod position. Both these measurements were made with identical rod settings

Beginning Friday, October 24, three flux capsules were exposed in the C-48 facility at 50, $83\frac{1}{3}$, and

[1] NaF, 4 8 mole %, KF, 50 1 mole %, ZrF_4, 41.3 mole %, UF_4, 3 8 mole %

[2] G. W. Keilholtz *et al*, *Solid State Quar Prog Rep May 10, 1952*, ORNL-1301, p. 20.

100% of reactor power, respectively. These will provide additional points on the power vs. flux curve.

After the MTR had reached full power, the power was decreased to N_L (1% of full power) to allow insertion of the salt capsule. Only 2.5 min was allowed for insertion and hookup, and full power was resumed in steps of 3 or 4 megawatts over a period of 24 minutes. This was a minimum time for this type of operation with xenon equilibrium conditions.[3]

The capsule was maintained at 1500°F for 116 hr at full flux. At this time (October 30), the reactor was shut down for fuel element change. The capsule was withdrawn and shipped to ORNL for salt analysis and metallographic examination The salt (0.39 g) dissipated 1900 watts/cc (288 watts total). The composition was ZrF_4, 70.1 wt %, NaF, 19.1 wt %, and enriched UF_4, 10.8 wt %.

DYNAMIC CORROSION TESTS

J. G. Morgan H. E. Robertson
A. E. Richt P. R. Klein

The design for the dynamic corrosion tests in hole HB-2 of the LITR and the fabrication of capsules have been completed [4] The plug that contains the rocking mechanism is near completion, and bench tests will begin in November. The first in-pile run will be a flux measurement along the length of the capsule to determine the effectiveness of the boral shield Instrumentation has been assembled and tested to enable the rocking cycles to be timed and counted. A platinum heater will furnish the additional heat necessary to maintain the fuel in the hot end of the capsule at 1500°F with a heat dissipation of 160 watts/cc from the salt.

STATIC CORROSION

D. D. Davies C. C. Webster

Three Inconel capsules, each containing 0 7 g of zirconium-base salt of composition No. 27, were irradiated for 140 hr at 140 watts/cc in the C-48 facility of the LITR. Neutron-flux level averaged about 2.3×10^{13}. Two bench tests were conducted under the same conditions as those of two of the irradiated capsules.

A series of long-term runs were initiated to study the effect of an extended irradiation on the chemical stability of the fuel and the corrosion aspects of the Inconel containers. Two capsules were exposed in the C-48 facility of the LITR but were removed after 565 hr because of a heater failure. The two capsules now being irradiated have run about 420 hr, and it is hoped that the test can be extended to at least 1000 hours. Bench tests of these two long-term runs are being conducted.

None of the capsules thus far irradiated have been opened for chemical and metallographic examination. Examination will be made as soon as the remotely controlled metallurgical facilities become available.

All capsules for these tests, as well as those to be used in the MTR and in the dynamic tests of the LITR, have been filled with fuel No. 27 supplied by the Materials Chemistry Division at Y-12. Filling and welding were accomplished under a helium atmosphere in a recently installed dry box fitted for these operations.

RADIATION EFFECT ON THE MELTING POINT OF LIQUID FUELS

C. C. Webster

From physical observation, it appears that there is more than one melting point for the No. 27 fuel after irradiation. Since the fuel must flow through heated tubes, it is necessary to know the effect of irradiation on the melting point of the proposed reactor fuel Equipment has been developed to determine the effect of irradiation on the melting point.

Because of oxidation and handling difficulties, the melting point measurements must be made on small quantities of fuel (0.3 to 0.7 g) in the capsule in which exposure occurred. For the LITR irradiations, this means using a capsule of 0.450-in. OD and 0.135-in. ID with a 0.014-in.-wall thermocouple well in the center of the fuel. For the MTR irradiations, the capsule is 0.200-in. OD and 0.100-in. ID with no thermocouple well. In the latter case, the thermocouple must be spot welded onto the outside of the capsule in the hot cell.

Commercial furnace forms wound with Nichrome wire coils are available with an inside diameter of $2\frac{3}{8}$ inches. It was decided to put two capsules as near as possible to the center of a solid copper

[3] W. W. Pennington, *Materials Testing Reactor Prog. Rep Sept. 1952*, IDO-16034.

[4] D. F. Weeks, P. R. Klein, J. G. Morgan, H. E. Robertson, and A. E. Richt, *Solid State Quar Prog Rep. Aug 10, 1952*, ORNL-1359 (in press).

cylinder placed inside this furnace. Such a system seemed workable because of the low thermal conductivity of the capsule material relative to the copper. The large copper cylinder serves to heat the two capsules (one irradiated and one unirradiated) uniformly at the same rate and temperature. Two furnaces were constructed as shown in Fig. 7. The copper cylinders differed slightly to permit postirradiation handling of the two types of capsules used in the MTR and LITR static tests.

The bottom guard cylinder and the top guard cylinder, together with the mica and asbestos insulation, serve to reduce the heat loss from the main cylinder by conduction and radiation The firebrick on top reduced the heat leak from the capsule.

Four thermocouples, placed inside the capsule wells, were found to remain within 1°C of each other at the proposed rate of heating

A Leeds & Northrup program controller with a Leeds & Northrup Speedomax was used to control the heating and cooling cycle. The control thermocouple was of chromel-alumel and was peened into a hole in the center of the copper cylinder. The copper cylinder was heated at the rate of 5°C/min, allowed to soak for 6 min at the maximum temperature, and then cooled at the rate of 5°C/min.

The temperature of the unknown capsule is recorded by one pen of a two-pen Brown recorder that has a full scale range of 2.5 millivolts. A differential thermocouple is used between the unknown and the standard. Any change in the reading of the differential thermocouple is recorded by the other pen of the two-pen Brown recorder that has a full scale range of 1 millivolt. A sample of the resulting chart is shown in Fig. 8.

All thermocouples used on the specimens were chromel-alumel and made from No. 30 wire. The thermocouple wiring diagram is shown in Fig. 9 Because the Brown recorder is a null instrument, it is possible to use the thermocouple wiring shown without interaction between the couples.

To date, the equipment has been bench tested by using sodium chloride as the standard and 0 6 g of the unirradiated fuel as the unknown. The results are shown in the accompanying graph (Fig 8).

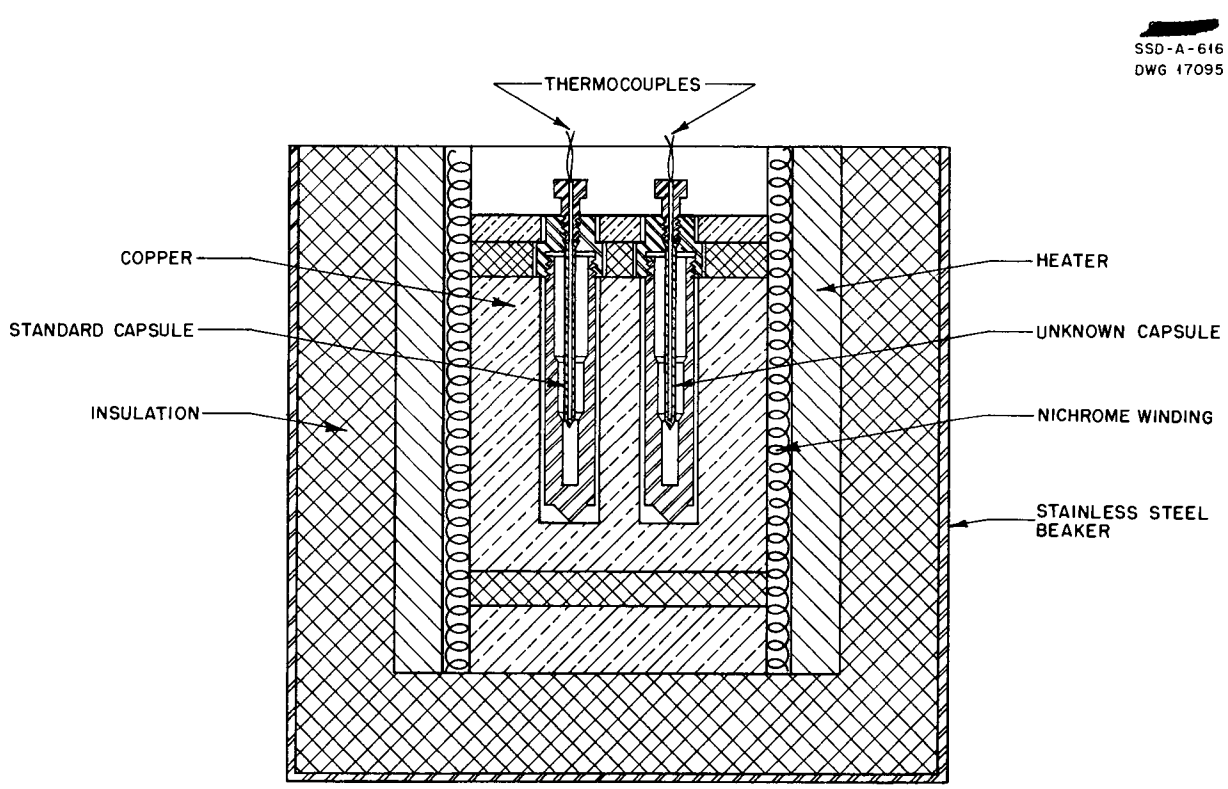


Fig. 7. Furnace for Determination of Melting Points of Irradiated Liquid Fuels.

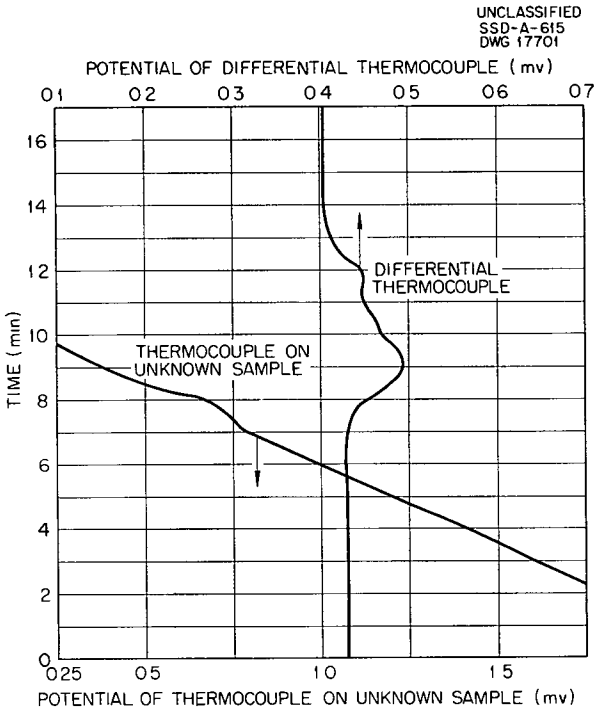


Fig. 8. Typical Strip Chart for Melting Point Determinations.

The 2 5-mv scale had five different ranges that could be used by switching from one range to the next.

The equipment is now being prepared to run melting-point curves of irradiated fuel within the hot cell.

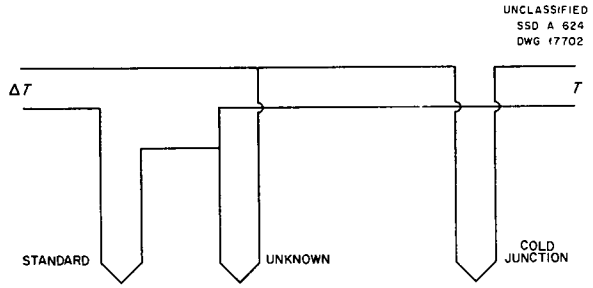


Fig. 9. Thermocouple Arrangement for Determination of Melting Point for Irradiated Liquid Fuels.

MOLECULAR CRYSTALS

M T. Robinson G. E. Klein

Work on the unit cell constants of copper phthalocyanine has been completed. The results have been accepted for publication in *J Am Chem Soc.* **74** (1952). The crystal was found to be tetragonal with a_0 = 17.367 Å, c_0 = 12.790 Å, Z = 6 ($C_{32}H_{16}N_8Cu$), d_{calc} = 1.488 g cm^{-3}, and d_{obs} = 1.49 g cm^{-3}. The probable space group is C_{4h}^1 – *P4/m*. These results are based on 29 x-ray diffraction lines. The unit cell constants are essentially the same as those reported previously.[5] The improved results obtained here allowed more certain assignment of the space group

[5] M. T. Robinson and G. E. Klein, *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

CRYSTAL PHYSICS

RADIATION CORROSION OF LIQUID FUELS IN INCONEL

W. J. Sturm J. V. Hilyer[1]
R. J. Jones[1]

A final series of bombardments of fluoride eutectic fuels in Inconel containers has been completed for ANP. Two zirconium-containing fuels were studied, eutectics Nos. 21 and 27, each in 0 010-in. Inconel tubing and under 22-Mev proton bombardment. Examination of the targets was made for radiation-induced effects on fuel stability and on the rate of corrosion of the Inconel container. In order to simulate the proposed aircraft engine application of these systems, efforts were made to duplicate the power densities and the temperature of the engine and to extend the duration of the bombardments as far as reasonably practicable. A limitation on input-power densities was established by the thermal conductivity of the target fuel element and the aircraft-engine design temperature as a boundary condition (815°C), a limitation on length of bombardment was imposed mainly by the stability of cyclotron operation, since a momentary increase in proton beam current was sufficient to melt the fuel pin.

In order to decrease the probability of target destruction by a surging proton beam, tandem

[1] Electromagnetic Research Division.

thermocouples were mounted on the target face. The outputs of these couples were monitored continuously on appropriate recording instruments and were interlocked with the cyclotron dee voltage in such a way that if the temperature of either thermocouple exceeded 860°C, the proton beam was instantly interrupted. In this manner, it was possible to extend the practical duration of the irradiation to nearly 100 hours.

Hitherto unreported results with the eutectic No. 21 are shown in Table 1. There is little, however, to indicate that a change in the earlier estimates is needed There is a fairly reproducible, but small amount, of radiation-induced increase in intergranular corrosion, which is found only in targets bombarded for periods in excess of about 4 hours. Also, what is more critical, radiation corrosion is apparently induced by proton bombardment only in energy dissipation fields in excess of about 3000 watts/cc.

The results of a completed series of tests with the newer No. 27 eutectic (NaF, 46.0 mole %, ZrF_4, 50 0 mole %, and UF_4, 4 0 mole %) are presented in Table 2. This series, irradiated with the interlocked thermocouple circuits, was extended to longer times without undue loss of targets because of overheating.

Again, results in this series show no radiation corrosion for energy dissipations below 3000 watts/cc for irradiations as long as 92 hours. The most significant corrosion in this group was found in the fuel element irradiated at 3300 watts/cc for 46 hours. The results of metallographic examination of this specimen are shown in Fig. 10. In addition to an intergranular corrosion layer of about 0 002 in. (Fig. 10a), for the first time in a proton-bombarded fuel element, a 0.0015-in.-deep layer was found inside the tube over the bombarded area of the Inconel. This layer (Fig. 10*b*), through observations of its magnetic properties with colloidal iron, was shown to be depleted in chromium. There is some indication of a double banding in the layer, but the colloidal-iron test showed no difference in magnetic properties across the layer, in these properties this phenomenon closely resembles the effects observed in static and circulating fuel bench experiments done on this fuel in independent metallurgical studies. It probably is not associated with the proton irradiation. The results of the test serve as an interesting indication of the sensitivity of the trace-element analyses, for despite the corrosion and chromium leaching seen metallurgically, little increase in the chromium content over that of other bombarded samples is seen

The 92-hr irradiation yielded results that, although somewhat unexpected, serve to indicate a strong dependence of radiation corrosion upon energy density. The dissipation rate for this bombardment was about 1700 watts/cc, and both chemical and

TABLE 1. RESULTS OF PROTON BOMBARDMENT OF EUTECTIC NO. 21 IN INCONEL CAPSULES

SAMPLE NO	DURATION OF IRRADIATION (hr)	POWER DENSITY (watts/cc)	FUEL ANALYSIS AFTER IRRADIATION					CAPSULE CORROSION* (in)
			Fe (ppm)	Ni (ppm)	Cr (ppm)	U (%)	F (%)	
910	3	2400	440	410	520	6 45	39 0	0 0005
907	3		270	300	1010	6 96	38 4	0 0005
1107	4 5	2300	450	90	590	6.7	37.8	0.0005
1101	4 5	Furnace control	110	75	1100	6 5	35 2	0 0005
911	7	4100	510	185	670	5 5	38 1	0 0015
905	7	Furnace control	140	29	1100	6 87	38 9	0 0005
1109	8	1150	610	105	670	7 7	36.7	0.0005
1102	8	Furnace control	110	25	1090	7 4	34 6	0 0005

*0 0005 in is the limit of detectability

TABLE 2. RESULTS OF PROTON BOMBARDMENT OF EUTECTIC NO. 27 IN INCONEL CAPSULES

SAMPLE NO.	DURATION OF IRRADIATION (hr)	POWER DENSITY (watts/cc)	FUEL ANALYSIS AFTER IRRADIATION					CAPSULE CORROSION* (in.)
			Fe (ppm)	Ni (ppm)	Cr (ppm)	U (%)	F (%)	
1220	5.6	2800	1650	280	1730	7.85	41.7	0.0005
1205	5.6	Furnace control	980	40	3000	**	**	0.0005
1214	10.6	2200	1780	55	1730	7.94	40.8	0.0005
1203	10.6	Furnace control	***	***	***	***	***	0.0005
1217	14.3	2000	2100	30	1310	7.34	**	0.0005
1204	14.3	Furnace control	***	***	***	***	***	0.0005
1215	15.4	2900	1530	65	1640	7.53	41.3	0.0005
1206	15.4	Furnace control	460	75	3400	**	**	0.0005
1221	46.4	3300	3500	230	2100	7.28	40.2	0.002
1201	46.4	Furnace control	1250	180	2700	**	**	0.0005
1219	92	1700	1950	50	1660	7.70	40.6	0.0005
1202	92	Furnace control	***	***	***	***	***	0.0005

*0.0005 in. is the limit of detectability.

**Insufficient sample for analysis.

***Analysis not yet completed.

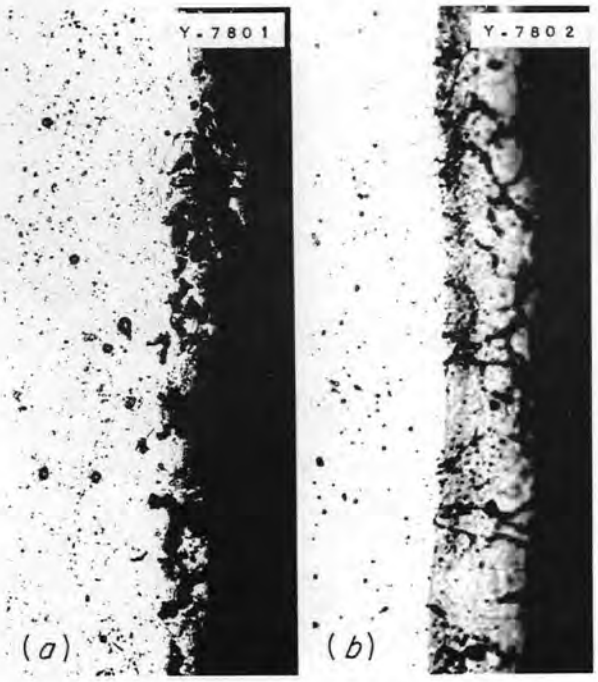


Fig. 10. Corrosion in Proton-Bombarded Inconel Container.

metallurgical examination showed no corrosion caused by the proton beam. Assuming this particular variation of eutectic composition does not alter radiation corrosion rates significantly (previous experience indicates this is probably true), the indications are that the corrosion rate depends strongly upon power density. It might be added that because the depth of corrosion in all cases observed is but a few thousandths of an inch regardless of the length of the irradiation, it appears that the corrosion rate quickly decreases from its initial high value.

In the course of the metallurgical examination of the 92-hr specimen, examination was made of corrosion at the point of a thermocouple weld. Because the structure at the weld was essentially that of cast Inconel and since no evidence of corrosion was found, there is some indication that cast Inconel is not much less resistant to radiation corrosion than is the more highly fabricated material.

PROTON BOMBARDMENT OF ALUMINUM

D. Binder

Neutron bombardment, even exposures as high as 10^{20} nvt, has little or no effect on aluminum.

Aluminum under long exposures, such as in slug casings, has shown some changes Proton bombardment, on the other hand, seems to produce a marked change in properties that can be detected most conveniently by hardness measurements. The exact cause of the changes is not known. Accordingly, two blocks of 99.9%-pure aluminum were subjected to an irradiation on the order of 100 μa-hr of 22.5-Mev protons. The blocks were held in a water-cooled target chamber behind a 12-mil aluminum window that reduced the incident energy to 21 Mev. The blocks were tilted with respect to the beam so that the protons near the top face entered the front face and emerged through the top face

The top face showed the range of the protons clearly, as a line appeared at about the correct distance from the front face. Hardness traces of the top face were taken before and after irradiation as a function of distance of penetration of the beam, and a typical set of data is shown in Fig. 11 Despite a scatter in the points, a peak is evident that corresponds approximately to the range for 22.5-Mev protons. The discrepancy is about 1 Mev (correction had been made for the window), and because the error in the measurement of beam energy is about 0.5 Mev, this is good agreement. The question remains was the hardness change caused by the trapping of hydrogen atoms or displacement of aluminum atoms? To answer this question, annealing experiments are planned that will use Continental Carbon Co. precision resistors

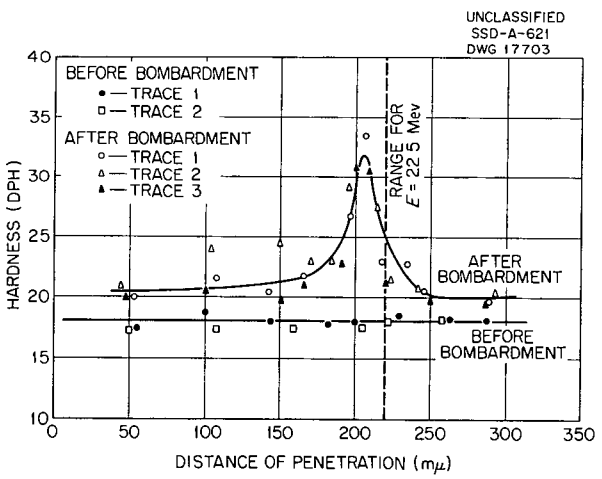


Fig. 11. Effect of Proton Bombardment on Hardness of Aluminum.

as hydrogen detectors.[2] These resistors are made of PdO, which is reduced to Pd by the hydrogen, thus effecting a large reduction of resistance from that of a semiconductor to that of a metallic conductor. A preliminary test on an available 1-megohm resistor in a leaky chamber filled with hydrogen showed that the resistance decreased to 200,000 ohms. Careful measurements will be taken to determine rates of decrease in resistance vs pressure.

METHOD OF MEASURING DIELECTRIC CONSTANT

D. Binder R A Weeks

The behavior of the input impedance of a quarter-wave line near the quarter-wave frequency indicates a convenient method for measuring the dielectric constant and attenuation factor of insulators in cables. Consider a line of length x terminated by an impedance Z_x. The impedance of the line is

$$Z_{in} = Z_0 \frac{Z_x + Z_0 \tanh \gamma x}{Z_0 + Z_x \tanh \gamma x}, \qquad (1)$$

where Z_0 is the characteristic impedance of the line and γ is the propagation constant

$$\gamma = \alpha + i\beta = \alpha + i\frac{2\pi f}{v},$$

where α is the attenuation factor, f is the frequency, and v is the velocity along the line At the quarter-wave frequency, $\beta x = \pi/2$ If attenuation is neglected for the moment,

$$Z_{in} = Z_0 \frac{Z_x + Z_0 \tan \pi/2}{Z_0 + Z_x \tan \pi/2} = \frac{Z_0^2}{Z_x}$$

The impedance is inverted by the line but is purely resistive for a purely resistive load at the quarter-wave frequency. Since the velocity along the line equals $c/\sqrt{K}$, where K is the dielectric constant and c is the velocity of light, the quarter-wave frequency is

$$f_{1/4} = \frac{c}{4\sqrt{K}\, x},$$

and changes in dielectric constant can be measured with half the precision of the quarter-wave frequency

(2) B. R. Gossick, *Palladium Film Fast Film Neutron Detector*, ORNL-650 (April 11, 1950).

Examination of the behavior of Z_{in} for frequencies slightly larger or smaller than $f_{1/4}$ reveals a method for both a precise measurement of the quarter-wave frequency and a less precise concurrent determination of the attenuation factor. For small Z_x/Z_0 and small αx,

$$\frac{Z_0}{Z_{in}} = \frac{Z_x}{Z_0} + \alpha x + i\pi \frac{\Delta}{2}.$$

Where Δ obeys the relation

$$\beta x = \frac{\pi}{2}(1 + \Delta), \qquad \Delta = \frac{df}{f_{1/4}} + \frac{1}{2}\frac{dK}{K}$$

If, then, the admittance $1/Z_{in}$ around $f_{1/4}$ is measured, the complex part, or susceptance, will change from positive to negative on passing through $f_{1/4}$ and will allow a precise determination of $f_{1/4}$. Any change in $f_{1/4}$ (and therefore also any change in K) can then be detected with precision Also, a change in attenuation may be detected independently as a change in the real part, or conductance, which does not vary with frequency near $f_{1/4}$.

Some time ago, a series of measurements was made that serves as a test of these ideas.[3] A quarter-wave line for an operating frequency of about 10 mc was cut from RG 11/U coaxial cable, and a small copper wire was soldered to the two terminals at one end. The other end was connected to the "unknown" arm of a General Radio Co. Twin-T bridge. The admittance was measured from 9.2 to 10.2 mc, and $f_{1/4}$ was determined as 9 74 mc with a probable error of 0.1%. This allows changes in K on the order of 0 2% to be detected. Also, the conductance was measured as 348 μmhos and varied by 5% or less on changing f. Then $Z_x/Z_0 \sim 0.03$, and from equation (1), an increase in αx from 0 to 0.03 would double the measured conductance. This test case is then suitable for an in-pile test of the effects of irradiation on α and K in commercial cable. The quarter-wave line may be coupled to the outside by a half-wave line, with the result that the signal is the same but inverted, also, some undesirable attenuation will be introduced. Several further tests of the method will be made.

REMOTE METALLOGRAPHY

M. J. Feldman R. U. Acton

The design and fabrication of all the major pieces of equipment for the mechanical phases of metallography-specimen preparation have been completed. A number of metals has been bench tested on the integrated process, and actual remote polishing of nonirradiated samples is now in process.

The metallographic process equipment is housed in cell No. 2 of the Solid State Building. A floor plan of this cell is shown in Fig. 12. Two Tell manipulators are available for use in this cell. The metallographic equipment is on the left side of the cell as the cell is faced from the operating area. Transfer drawers from the two adjoining cells provide facilities for transfer of samples from auxiliary operation. The equipment, as seen from the right side of the cell, is shown in Fig. 13.

The remote cut-off machine will handle a bulk sample up to 18 in. length and up to 1½ in. in diameter (or square). For samples larger than these dimensions, use will be made of the machine shop in cell No. 1 to reduce the sample size. The cut-off wheel leaves the sample with a surface that can be handled by the Lapmaster.

The samples are mounted in a 1¼-in. bakelite mount. The mounting press was designed at ORNL and incorporates facilities for the addition of powder bakelite and for the addition of identification disks

[3] Unpublished memorandum from D. Binder to R. S. Livingston, April 23, 1951.

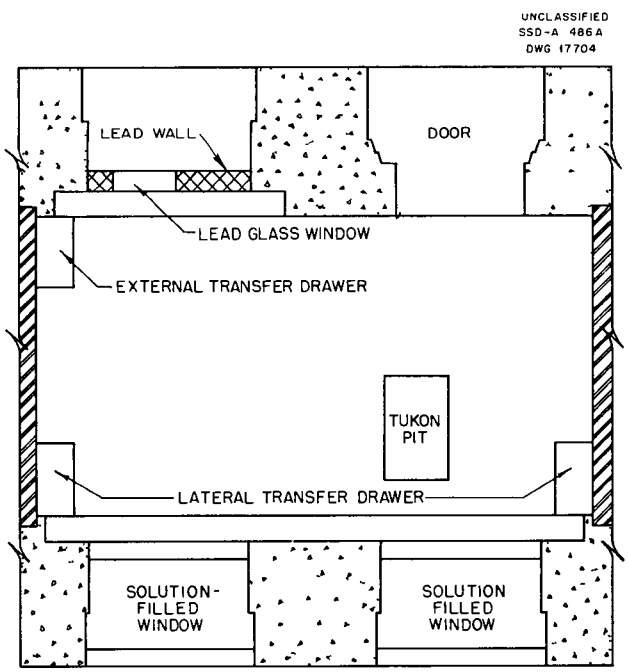


Fig. 12. Plan View of Cell No. 2, Building 3025.

of $^{15}/_{16}$-in. diameter soft steel to the upper face of the mount. The identifying disk is also used for magnetic gripping of the samples.

The Lapmaster, shown in Fig. 14, is a commercial production lapping machine that has been converted for remote operation. The Lapmaster is fed with 800-mesh alumina lapping compound. Running times in this step of the process vary from 5 to 30 min, depending upon the material. The Lapmaster will also serve as the mechanical preparation step for the electrolytic polishing and etching stages that are in the process of design and fabrication.

Following preparation on the Lapmaster, the samples are placed in polishing machines. Three Buehler low-speed machines have been converted for remote operation. The converted machine is shown in Figs. 15 and 16. The first machine utilizes 6 to 12 μ diamond dust and a microcloth-covered wheel. The second wheel operates with 1 to 6 μ diamond on a lead lapping wheel and is used as an intermediate polishing step for the preparation of corrosion specimens where the prevention of edge rounding is essential. The final polishing is done on the third wheel with 0 to 1 μ diamond dust on a microcloth-covered wheel.

Cleaning of the samples between the polishing steps is one of the most critical problems in the polishing procedure. A specimen washer has been designed for this purpose. The specimen washer is in reality a very compact manipulator. Since the diamond paste covers all three surfaces of the mounted sample the washer must clean all these surfaces, Fig. 17 shows the washer. The clamps take the specimen from the right-hand side and move it under a rotating brush where the disk-bearing surface is cleaned in a spray of solvent. The sample is then transferred to a rotating magnetic holder where the sides and specimen surface are sprayed by two separate solutions and at two speeds. An air spray then dries the sample and the left-hand clamps remove it for the next step.

Figure 18 shows the etching manipulator in which use is again made of magnetic gripping. The sample is immersed in the etching solution for a predetermined length of time and then washed, alcohol rinsed, and air dried. After removal from the etcher, the samples are placed in the metallograph.

A standard Bausch & Lomb GBILS metallograph was installed so that the microscope stage is in the cell and the bellows and viewing screen are outside. A 12- by 20-in. lead glass window was installed in the 6-in. lead wall through which the metallograph is mounted. No attempts have been made to remotely change the objectives because it is felt that the magnification needed can be predetermined before the hot samples are introduced. The metallograph is shown in Figs. 19 and 20.

Table 3 shows the tentative procedures that have been adopted for a number of metals.

EFFECT OF LITR IRRADIATION ON THERMOCOUPLES

A. Foner Cohen L. C. Templeton

During the course of measuring the absolute thermal conductivity, K, of various metals and alloys in hole HB-3 of the LITR at temperatures from approximately 300 to 850°C, measurements of the temperature T were obtained, as well as the more frequently measured differential temperatures that were necessary to calculate K. Duplex chromel-alumel thermocouple wires were stripped of insulation and threaded through the small holes in tubular-lead Kovar seals. The leads were then individually insulated by ceramic tubes along their length from the Kovar seal to the hot junction at the sample. The atmosphere surrounding the couples was stagnant air. The cold junctions were maintained at constant temperature by means of a large ice bath that consisted of a mixture of distilled water and crushed ice that was made from distilled water.

A series of constant-temperature runs was made at various temperature levels. The temperatures were obtained by means of either an approximately constant flux level or a constant heater input. Thermocouple emf values were measured with a type K-2 potentiometer and a galvanometer and plotted as a function of time. It is therefore possible to follow the behavior of the thermocouples as a function of irradiation at various temperature levels. The maximum operating flux of the LITR was 1500 kw. The behavior of chromel-alumel couples at high temperatures as a function of time must, however, be taken into consideration. Work of this sort has been reported by A. I. Dahl,[4] who

[4] A. I. Dahl, "Stability of Base Metal Thermocouples in Air from 800 to 2200°F," *Temperature, Its Measurement and Control in Science and Industry*, p. 1238, Reinhold, New York, 1951.

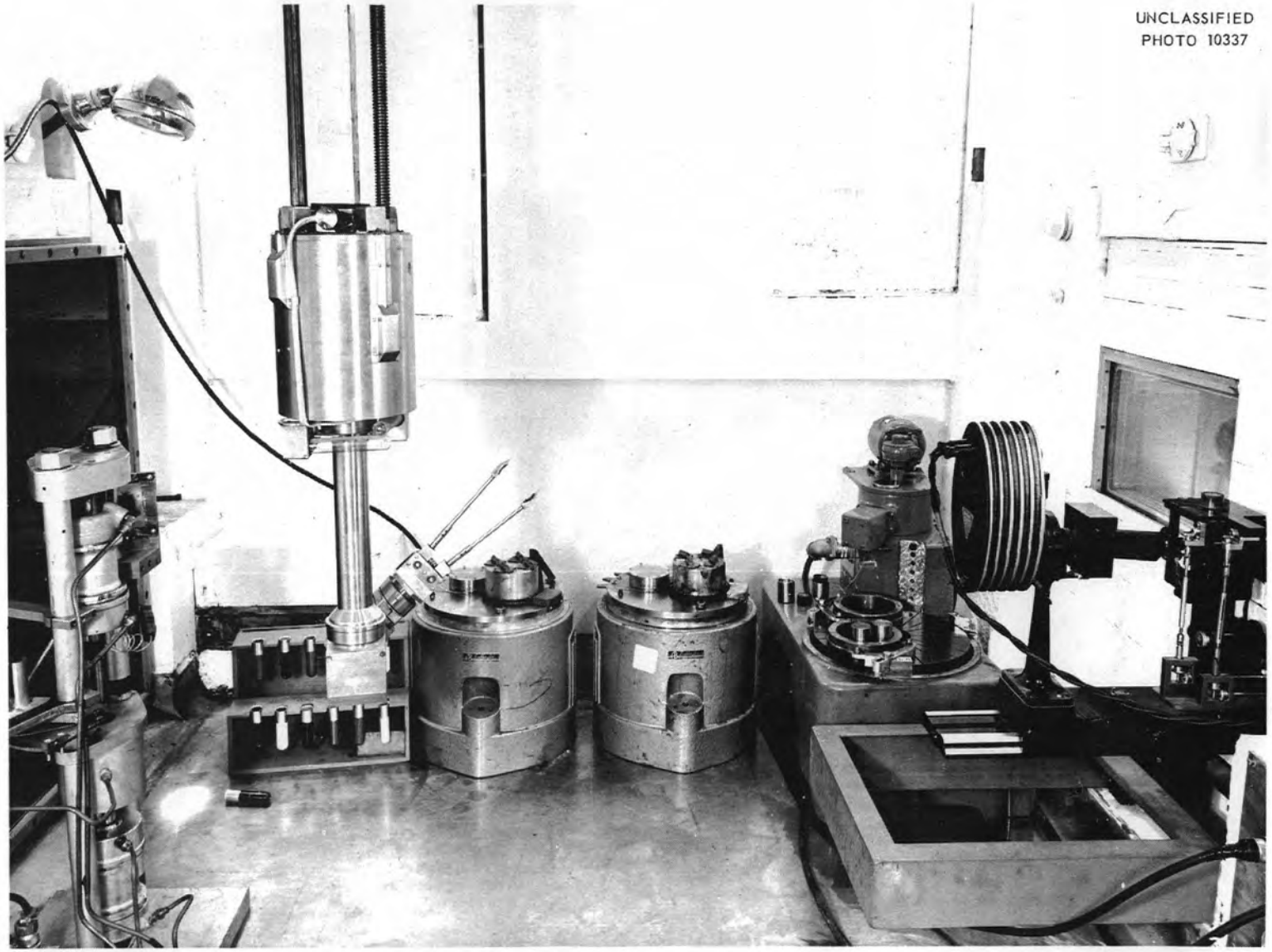


Fig. 13. Equipment in Cell No. 2, Building 3025.

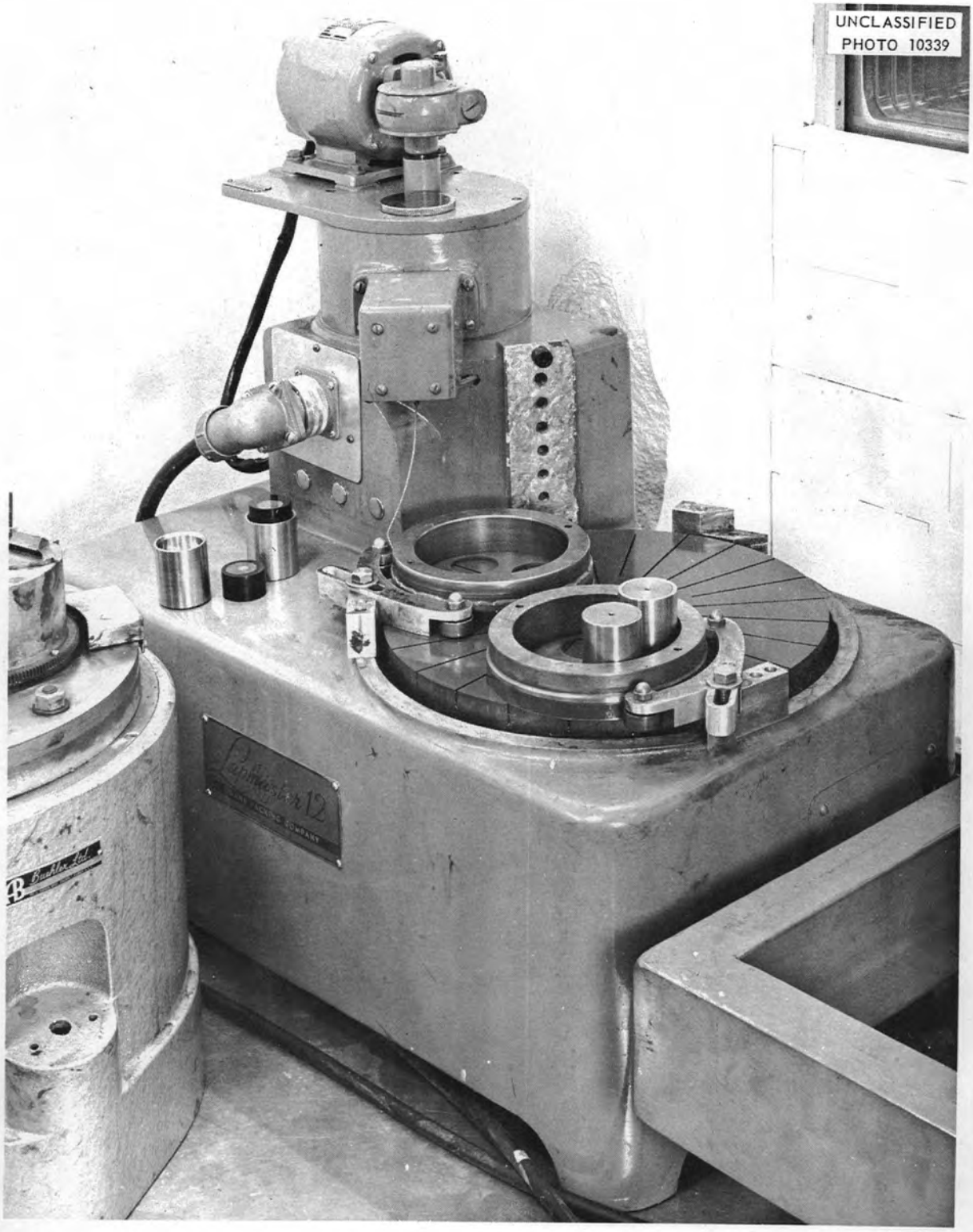


Fig. 14. Remotely Controlled Lapmaster.

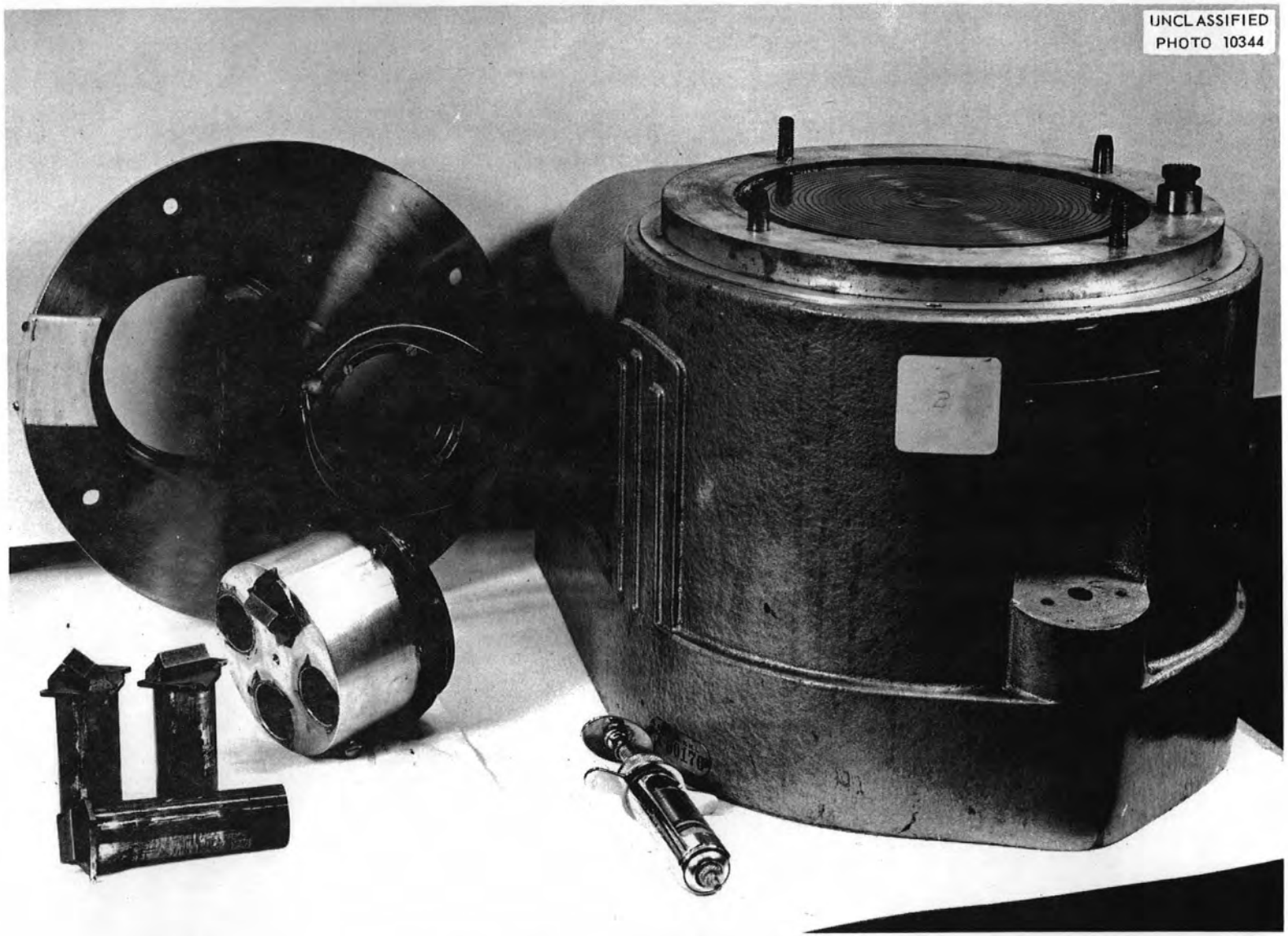


Fig. 15. Disassembled Polishing Apparatus.

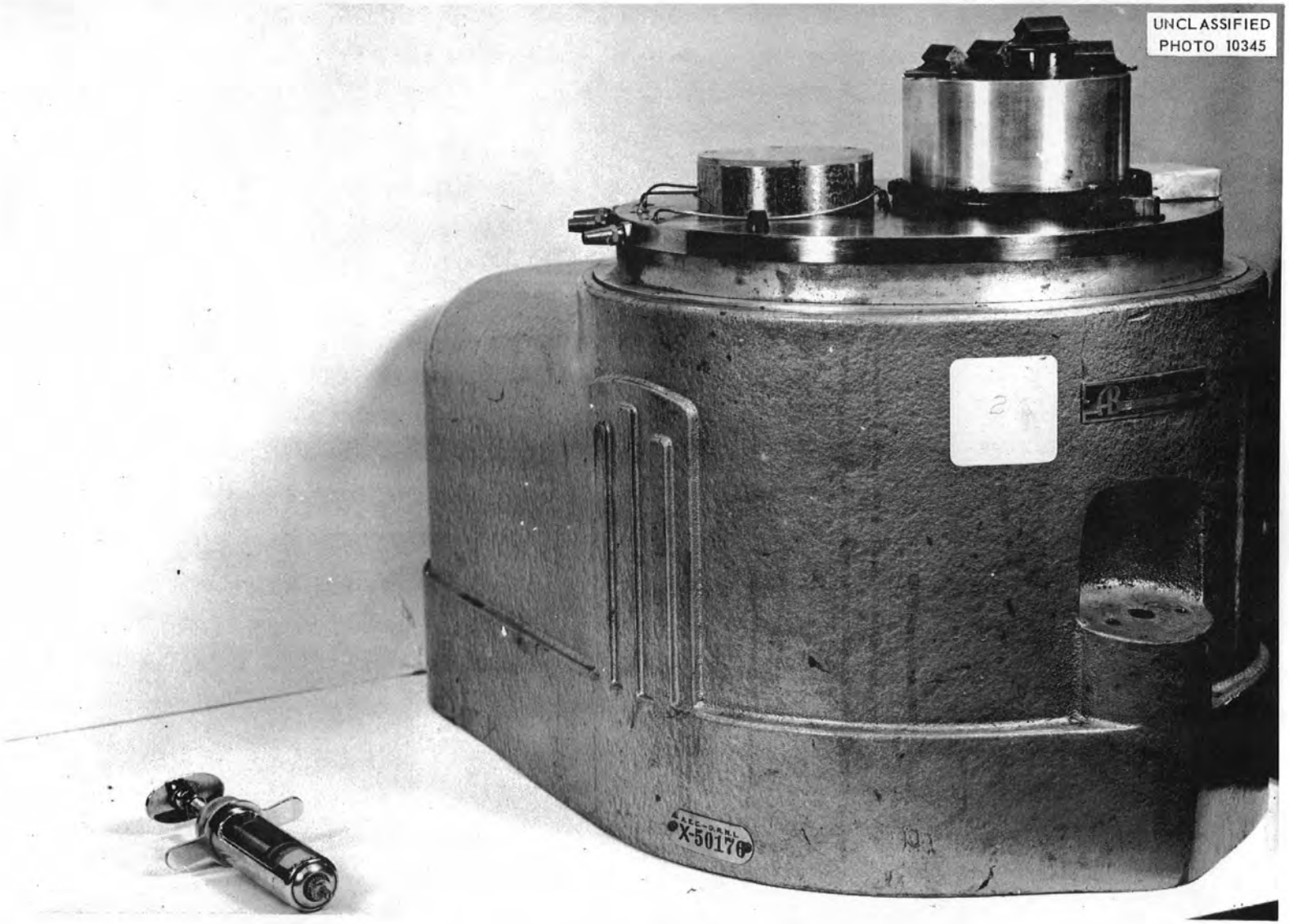


Fig. 16. Remotely Controlled Buehler Polishing Machine in Operation.

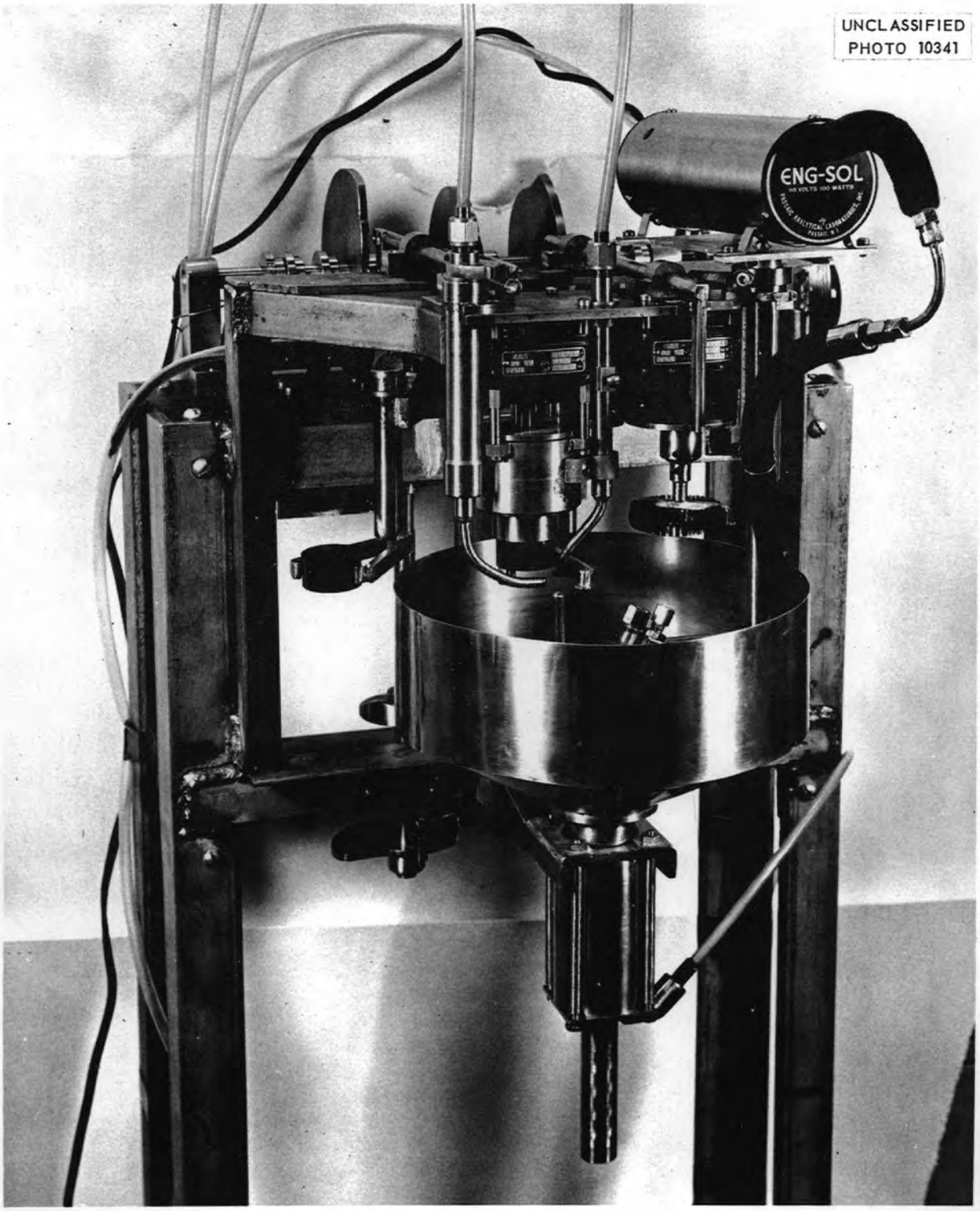


Fig. 17. Washer for Radioactive Specimens.

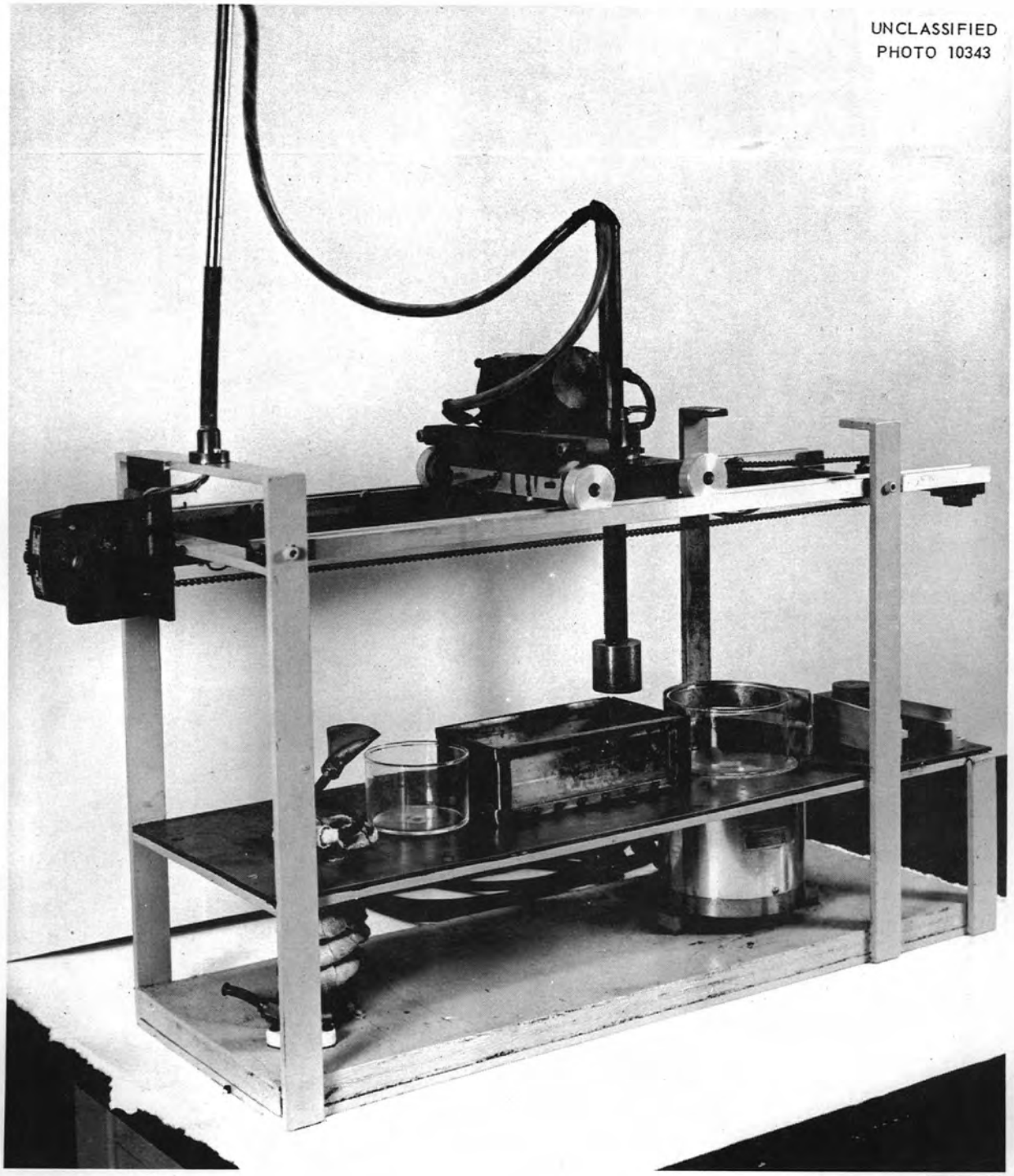


Fig. 18. Etching Manipulator.

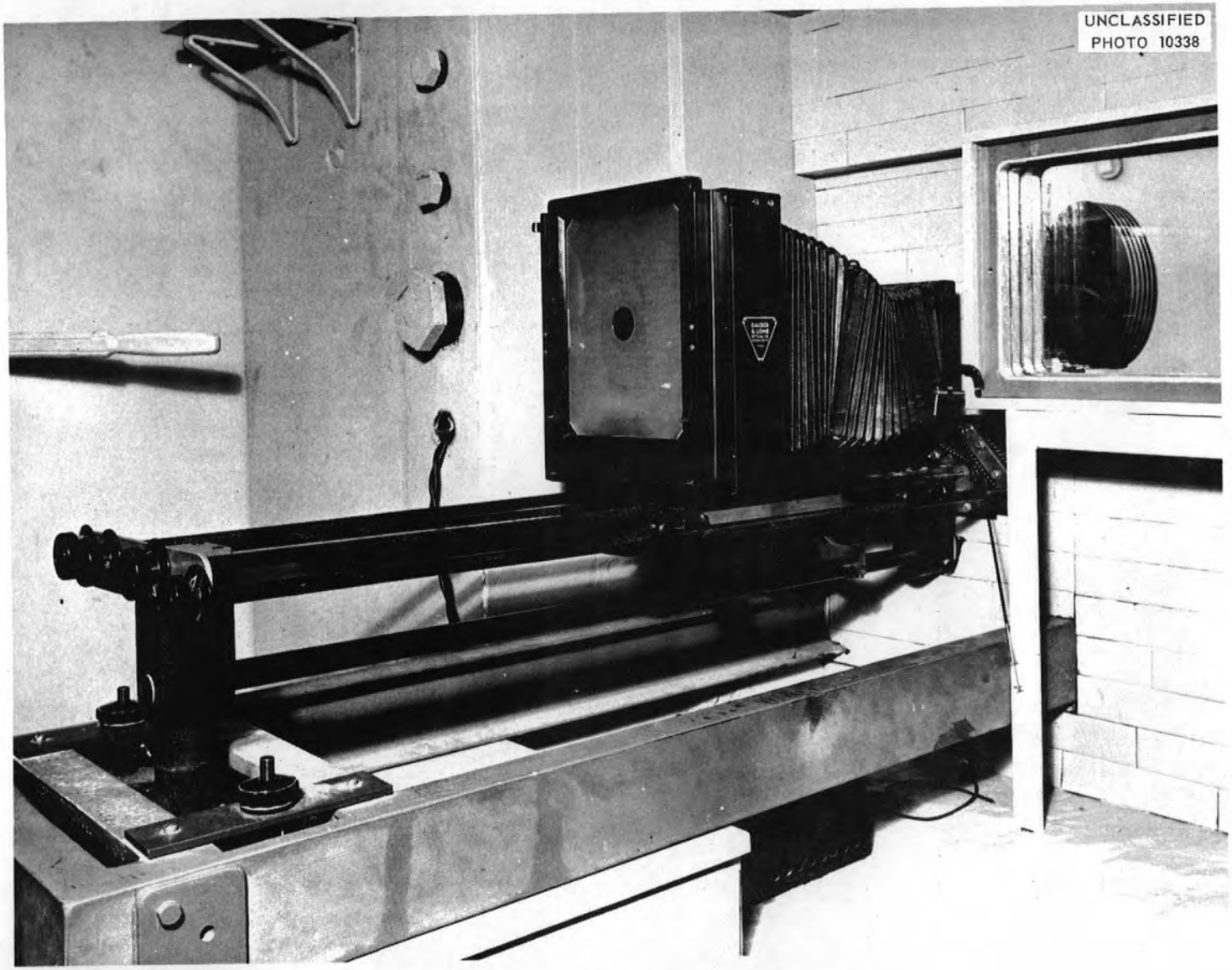


Fig. 19. Outside-Cell Portion of Metallograph.

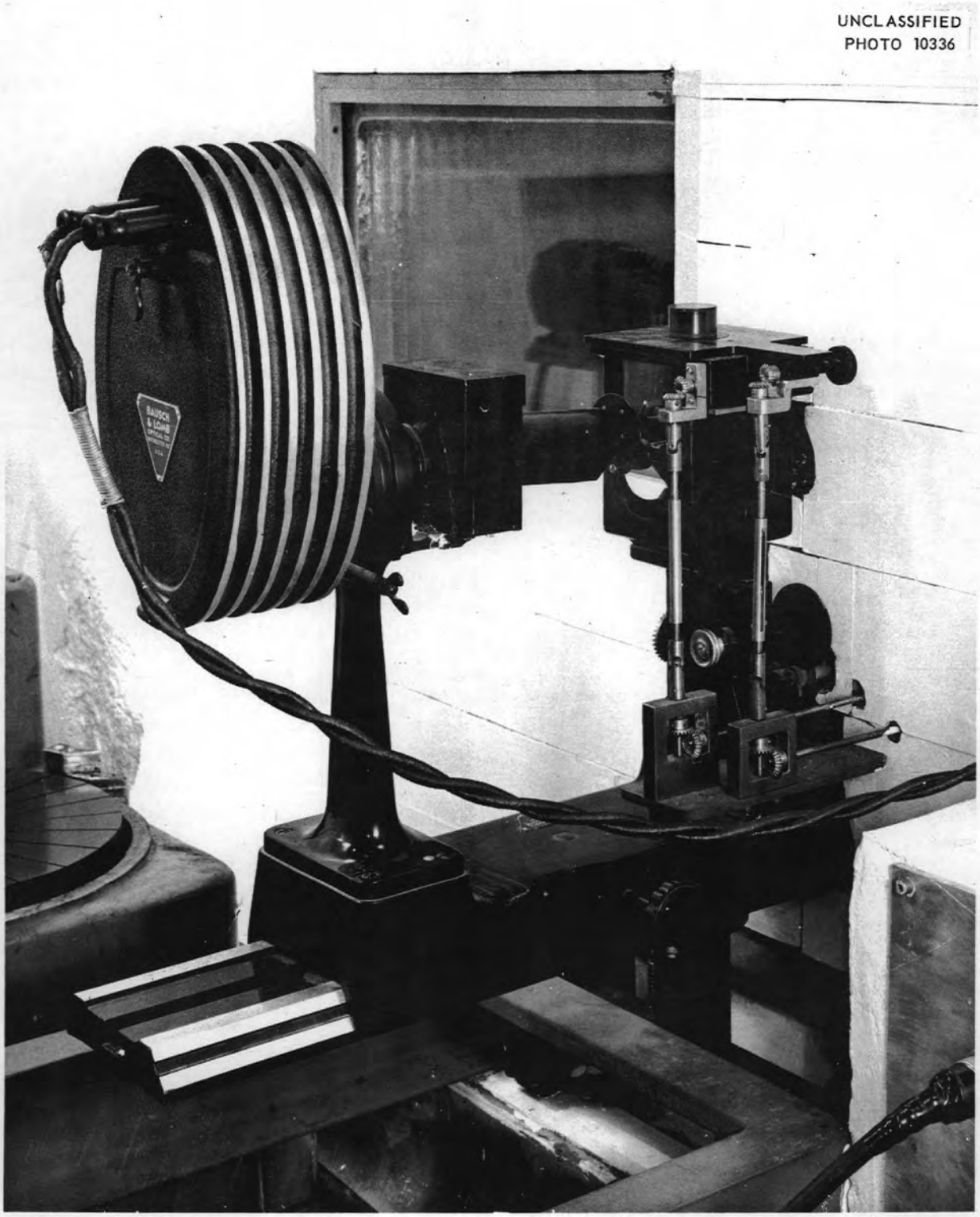


Fig. 20. Inside-Cell Portion of Metallograph.

has shown that the output emf of chromel-alumel couples that were left undisturbed at high temperatures showed appreciable calibration changes. These changes with time (increases in thermocouple emf for chromel-alumel thermocouples) are dependent on the temperature level and the wire size.

Figure 21 shows the output emf values of chromel-alumel thermocouples that were exposed in the LITR under the experimental conditions indicated. The data collected prior to the beginning of the in-pile portion of the experiment indicate slight changes in thermocouple emf that can be correlated qualitatively with the changes in heat power level.[5] The calculated heat power is proportional to the difference in the measured temperatures of the outlet and inlet water that flows through the LITR cooling system and the water flow rate. The heat power depends primarily on the neutron-flux level. The neutron-flux level is controlled to approximately 1%[6] during a running period (one week). The week-to-week variations in the setting of the neutron-flux level may be several times this amount if the loading of the reactor lattice is changed, or if the experiment load in the reactor is changed, renewal of fuel elements and adjustment of fuel elements also are responsible for changes in the neutron flux distribution. At a flux level such that the sample and thermocouple temperatures caused by gamma heating are in the neighborhood of 300°C, a 1% change in neutron flux would be equivalent to a 3°C change in sample and thermocouple temperature.

Data from an experiment that was run at a different time and with a different sample and thermocouples are shown in Fig 22. Gamma heating at LITR flux level of 1500 kw was the heat supply to the sample and thermocouples The emf variations of the chromel-alumel thermocouples over the first 100 hr of irradiation are correlated qualitatively with the heat-power fluctuations. In this period, it should be noted that the copper-constantan thermocouple variations are in agreement with those of the chromel-alumel T H. Blewitt and R E. Jamison recently performed an experiment in the ORNL graphite reactor that employed copper-constantan and iron-constantan thermocouples at the constant temperature of liquid nitrogen No

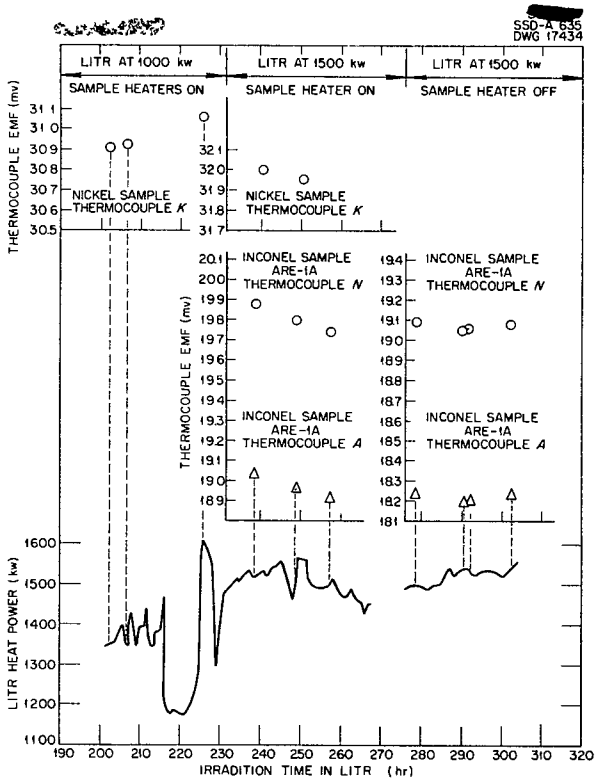
[5] For LITR heat and power calculations, see M. W. Belaga, R. J. Mulvihill, and I. S. Zonis, *Determination of Power Level for Low Intensity Training Reactor at Oak Ridge*, K-864 (Jan. 29, 1952).

[6] Estimated by C. Cagle of Pile Operations Dept.

TABLE 3. PROCEDURES FOR REMOTE METALLOGRAPHIC SAMPLE PREPARATION

MATERIAL	CUTTING[1]	POLISHING TIME (min)				ETCHING		NOTES
		Lapmaster with 800 Mesh Alumina	No 1 Wheel with Microcloth 6 to 12 μ Diamond	No 2 Wheel with Lead Lap [2] 1 to 6 μ Diamond	No 3 Wheel with Microcloth 0 to 1 μ Diamond	Etchant	Time (min)	
Copper		10	10	15	5 10	CrO_3 HNO_3 etc	2	(3 4)
Type 2S aluminum		10	10	10	10	1/2% HF	2	(4)
Molybdenum		20	20	20	30	Alkaline K_3FeCn_6	2	
Tungsten		40	30	20	30	Alkaline K_3FeCn_6	3	
Brass		10	10	10	10	Dilute NH_4OH	2	(4)
Type 24 ST aluminum		15	10	10	15	1/2% HF	2	(4)
Mild steel		25	20	15	15	Nital	2	
High carbon steel		30	25	20	30	Nital	2	
Type 310 stainless steel		30	30	15	20	Glyceral regia	2	(5)
Type 316 stainless steel		30	30	15	20	Glyceral regia	2	(5)
Type 347 stainless steel		30	30	15	20	Glyceral regia	3	(5)
Inconel		30	30	15	25	Glyceral regia	3	(6)

Notes
1. Testing not complete on use of fine diamond cut-off wheel.
2. Use of lead lap primarily to maintain flat corrosion face.
3. Most etching solutions used in very dilute form in order to lengthen etching time.
4. No. 2 wheel bypassed for structure examination.
5. By volume, 75% glycerine, 20% HCl, 5% HNO_3.
6. By volume, 50% glycerine, 40% HCl, 10% HNO_3.



COMPARISON OF LITR HEAT POWER WITH TEMPERATURES MEASURED BY CHROMEL ALUMEL THERMOCOUPLES

Fig. 21. Comparison of LITR Outlet-Water Temperature with Inconel Sample Temperature at 1000-kw LITR Power.

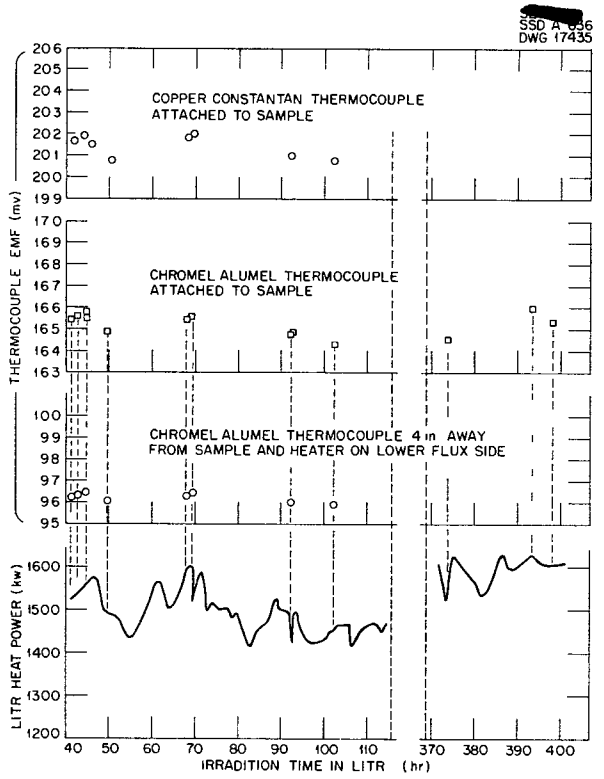


Fig. 22. Comparison of LITR Outlet-Water Temperature with Temperature of Two Samples.

change was found in the output emf over a total period of 60 hr, with a total nvt of $\sim 4 \times 10^{16}$ neutrons/cm^2 The accuracy of the measurements in the experiment would have revealed changes of approximately 1°C in the thermocouples More radiation damage to the copper-constantan should occur in the low-temperature experiment in the graphite reactor than at the higher temperatures ($\sim$400°C) in the LITR.

After the first 110 irradiation hours (see Fig. 22), the sample and thermocouples were subjected to higher temperatures for the next 260 hr (not shown in Fig. 22), including a temperature of 825°C for 130 hours. Following the irradiation of the specimens and thermocouples at 825°C, the experimental conditions were made the same as those existing during the first 110 hr (same flux level and no external heater) As shown in Fig. 22, the thermocouple emf in the period from 370 to 400 hr does not differ from the first 100-hr irradiation by an amount outside the limits of the variations ($\sim$1%) of the neutron-flux level. A slight reactor change that could cause a small change in the flux level was effected between the two periods plotted. The effect of aging the couples over the times and temperatures involved here are considerably smaller than the effect of neutron-flux variations. In any running period, damage to the thermocouples that would produce a change of thermocouple output emf of amount greater than that which can be explained by the estimated 1% change in temperature resulting from flux variation would have shown a systematic change in the thermocouple emf. Such systematic change was not observed in the period of the first 100 irradiation hours, nor in the period of 370 to 400 irradiation hours. The thermocouple emfs of the two periods are essentially the same, hence no systematic change seems evident over about 400 irradiation hours.

The results of another experiment are shown in Fig. 23, the neutron-flux level was 1000 kw. Comparison of the calculated heat power with

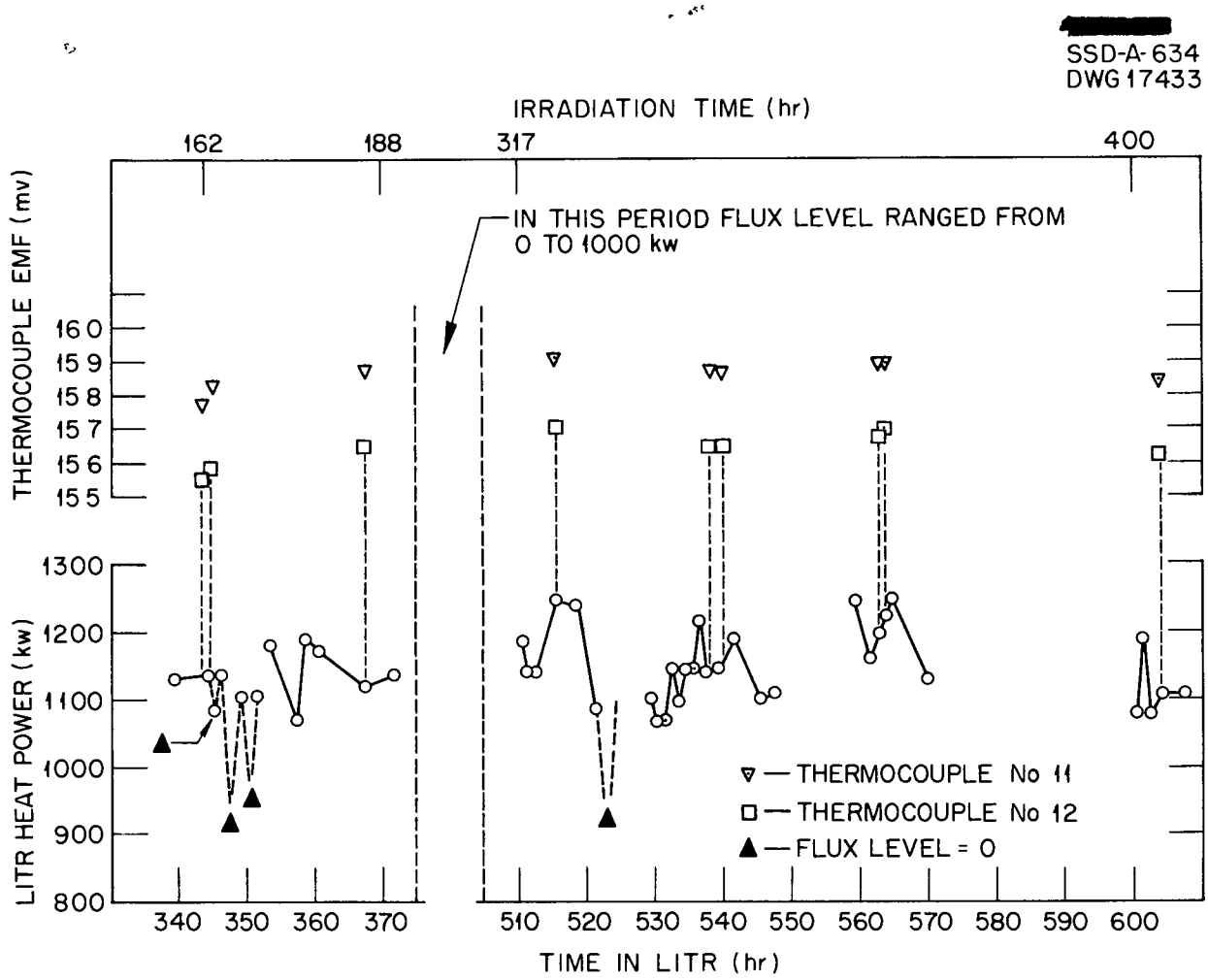


Fig. 23. Comparison of LITR Outlet-Water Temperature with Inconel Sample Temperature at 1500-kw LITR Power.

chromel-alumel thermocouple emfs is shown. The thermocouple emfs in the period from 317 to 400 irradiation hours do not show evidence of damage when a flux variation of ~1% at 375°C is taken into account. In the period from 188 to 317 irradiation hours, some reactor changes affecting the neutron-flux distribution in the reactor were made.

In conclusion, chromel-alumel thermocouples that were exposed in the LITR at neutron-flux levels that were maintained constant within the experimental limits (~1% in a given week) at 1000 and 1500 kw, show no damage produced by radiation. The maximum total irradiation time for an experiment at 1000 kw was 400 hr, for another experiment at 1500 kw, the irradiation time was 400 hours. The temperature at which the measurements in these two experiments were obtained was provided by gamma heating at the operating neutron-flux level. However, the thermocouple in the 1500-kw experiment was subjected to 130 irradiation hours at 825°C between the two periods of measurements compared.

HIGH TEMPERATURE THERMAL CONDUCTIVITY MEASUREMENTS ON UNIRRADIATED METALS AND ALLOYS

A. Foner Cohen

The construction of the apparatus in which the absolute thermal conductivity of various metals and alloys can be determined has been completed. The temperature range of the expected measurements extends from about 50 to 850°C or slightly higher if necessary. The apparatus has been heated up to 850°C in preliminary testing. An auxiliary recording circuit is being constructed to eliminate the laborious part of obtaining data.

NUCLEAR MEASUREMENTS

NEUTRON-SPECTRA MEASUREMENTS IN THE BSF

J. B. Trice F. J. Muckenthaler

The measurements of reactor neutron spectra by use of radioactivation methods are being extended to several new positions, two of which are in the BSF. Here it will be possible to compare the radioactivation results with those obtained from a proton-recoil counter used to measure neutron energy spectra in the same positions.[1] The reactor-fuel configuration is shown in Fig. 24. The position shown nearest the reactor corresponds to a neutron thermal flux of about 1.5×10^{12} neutrons/cm^2·sec at 100 kw reactor power. The position farthest from the center corresponds to a thermal neutron flux of 1.33×10^8 neutrons/cm^2 sec and is at the end of a 2-ft long by ½-in.-dia hollow aluminum tube used as a neutron collimator.

For these tests, an attempt is being made to use as many nuclear threshold reactions as time and the handling facilities will allow. Some of the reactions that have not been used extensively before include $P^{31}(n,\alpha)Al^{28}$, $Si^{28}(n,p)Al^{28}$, $Si^{29}(n,p)Al^{29}$, and $Cu^{63}(n,2n)Cu^{62}$, with energy thresholds at ~3, 5.5, 4.6, and 12 Mev, respectively. Of these four reactions, $Si^{28}(n,p)Al^{28}$ and $Si^{29}(n,p)Al^{29}$ have been observed during the present series of tests in the LITR. The $P^{31}(n,\alpha)Al^{28}$ reaction has not been observed during the counting of $Mg_2P_2O_7$, possibly because of the presence of 10.0-m Mg^{24} activity that may be strong enough to obscure the rapidly falling 2.3-m Al^{28} activity. For the $Cu^{63}(n,2n)Cu^{62}$ reaction, which is important because of its rather high threshold energy (~12 Mev), a small quantity of pure Cu^{63} was obtained from the ORNL Isotope Branch of the Operations Division. This isotope is being irradiated as CuO. To date, extensive irradiation tests in high intensity (~10^{12} neutrons/cm^2·sec) neutron fields have failed to yield the reaction product, Cu^{62}.

Another test to determine fast-neutron data was performed for the lattice configuration shown in Fig. 25. The test was a joint project with the Bulk Shield Testing group. The neutron measurements were made in order to obtain the value of the neutron flux above 10.0 Mev The Bulk Shield Testing group is using the information to assist in determining the (n,p) cross section for O^{17} above 10 Mev, which is approximately the threshold for the nuclear reaction. Aluminum-27 was used for the activation because it has two nuclear reactions that are conveniently located with respect to energy. The first of these, $Al^{27}(n,p)Mg^{27}$ with a 10-m decay period, has an effective threshold energy of 4.6 Mev in a fission spectrum, and the second, $Al^{27}(n,\alpha)Na^{24}$ with its 15-h decay period, has an effective threshold at 8.1 Mev. Previous experiments indicate that the shape of the fission spectrum remains well preserved above 4.0 Mev, even after the fission neutrons have penetrated several centimeters of moderating material. Since the present measurements were taken close to fissioning material, it was assumed that the neutron spectral shape was unaltered. Therefore only two positions on the energy scale, that is, those at 4.6 and 8.1 Mev, were used. The experimental arrangement is shown in Fig. 26, which is a diagram of the strip used for mounting the material to be irradiated. For irradiation, the strip was located at position 27 in the reactor assembly. The two outside cadmium-enclosed aluminum samples were

[1] Proton-recoil counter measurements made by R. Cochran (unpublished).

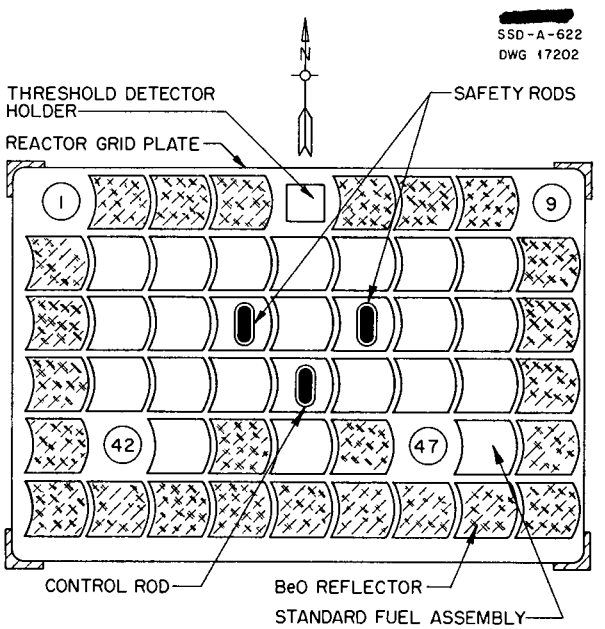


Fig. 24. Bulk Shielding Reactor, Loading No. 17.

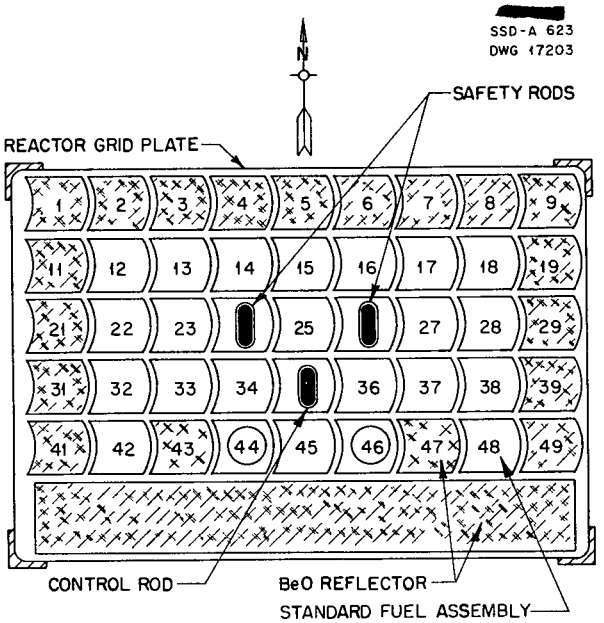


Fig. 25. Bulk Shielding Reactor, Loading No. 8.

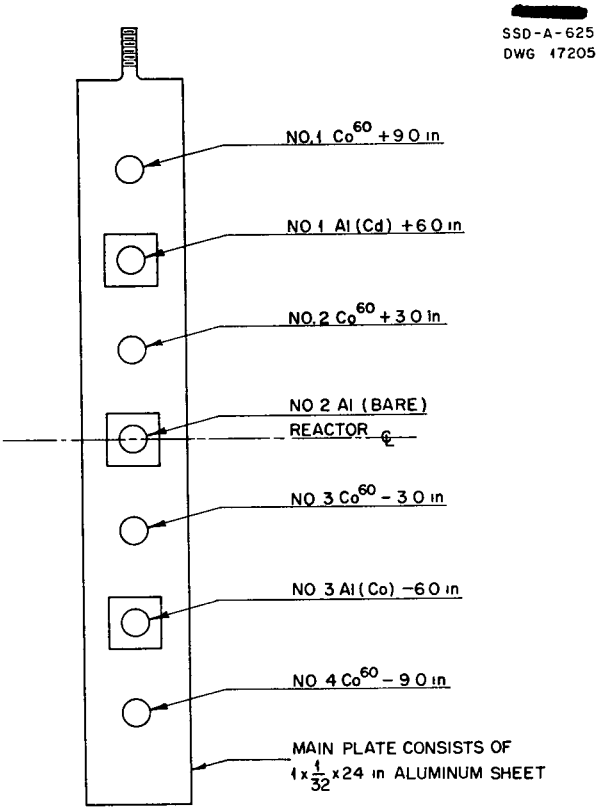


Fig. 26. Sample Holder for Test in Bulk Shielding Reactor.

located 6 in. above and 6 in. below the reactor center line. One bare sample of Al^{27} was located at the center point. The thermal flux was monitored by four Co^{59} strips located as shown in Fig. 26.

The neutron flux was found to be 1.68×10^{11} and 1.23×10^{10} neutrons/cm^2 sec above 4.6 and 8.1 Mev, respectively, at a position 6 in. above the center line. Corresponding numbers at 6 in. below the reactor center line were 1.63×10^{11} and 1.26×10^{10} neutrons/cm^2 sec. The average ratio of neutrons above 4.6 Mev to those above 8.1 Mev was found to be 0.075. For the fission spectrum according to Watt,[2] this ratio is 0.077. Comparison of these ratios indicates that the as-measured data fit the fission spectrum very well. On this basis, the total number of fission neutrons is 2.4×10^{12} neutrons/cm^2 sec. The thermal flux at the two positions averaged 8.1×10^{11} neutrons/cm^2·sec, and the reactor power was 100 kw. It is thought that experimental errors do not exceed 5% for actual measured fluxes.

[2] B. E. Watt, *Energy Spectrum of Neutrons from Fissions Induced by Thermal Neutrons*, LA-718 (Dec. 17, 1948).

IRRADIATION FACILITY FOR MTR

J. B. Trice F. J. Muckenthaler

In order to measure the fast-neutron energy spectrum for use with stress-corrosion measurements in hole HB-3 of the MTR, a rig is being designed that will include the following features

1. Pneumatic insertion and withdrawal of radioactivants will speed up the actual irradiation mechanism over the methods used previously in hole HB-2 of the LITR. This, together with automatic transfer of hot material to cold containers while inside the plug, will eliminate the hazards associated with manual transfer and will minimize the time spent in transporting the material to the counters. The data for short-lived materials will be improved because the delay time between the reactor and the counters frequently results in the

short-lived activities being almost obscured by those with longer lives.

2. The plug will be water cooled, a precaution that will be necessary in order to prevent melting of cadmium containers in the high-intensity gamma field.

3. An instrument, either a boron thermopile or a thermal-neutron fission chamber, will be permanently fixed into the plug so that a continuous power level record can be obtained.

4. Fission chambers, to be manually inserted into the plug, will measure neutrons at the same positions as those used with the radioactivants. At least three different kinds of fissionable materials will be used. Uranium-238, which has an effective energy threshold at about 1.6 Mev, Np^{237}, which has an effective threshold at about 1.0 Mev, and Th^{232}, which has an effective threshold at about 2.0 Mev for pile neutrons, will greatly strengthen the spectral data obtained with the radioactivants. The fission cross sections of these elements are not only more accurately known, but in general, they approximate true step functions more closely than the other threshold elements.

5. The plug is being designed with one or more floodable chambers in the vicinity of the highest radiation fields so that a measurement can be made of the effect that moderation has on the spectral form.

In addition to the radioactivants and fission chamber measurements, nuclear heating measurements throughout the plug will also be made.

NUCLEAR HEATING

J. B. Trice F. J. Muckenthaler

A calorimeter[3] was used to measure nuclear heat generated in type 347 stainless steel placed along the axis of hole HB-2 in the LITR. Calibration was effected by use of an electrical heater inserted into the calorimeter. Known rates of input electrical energy were then measured as a function of calorimeter wall temperatures, and a calibration curve was obtained.

Figure 27 shows nuclear heat production rate as a function of position along hole HB-2 in the LITR for an operating reactor power of 1.5 megawatts. These data are in agreement with previous estimates. The accuracy for any individual point is probably better than ±10%

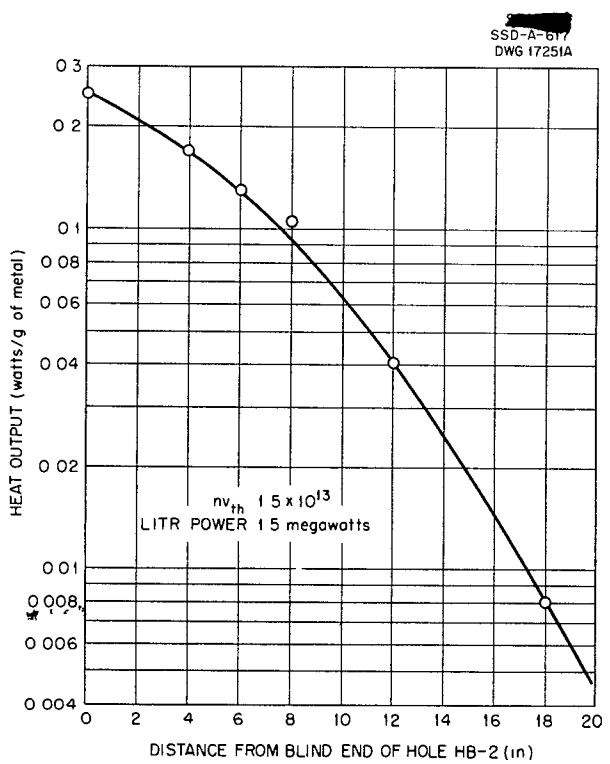


Fig. 27. Nuclear Heat Production for Type 347 Stainless Steel in Hole HB-2 of the LITR.

[3] J. B. Trice and D. T. James, *Solid State Quar. Prog Rep May 10, 1952*, ORNL-1301, p. 19.

SPECIAL PROJECTS

IRRADIATION OF MISCELLANEOUS MATERIALS

M. C. Wittels J. B. Trice

X-ray investigation of lattice transformations in diamond and cubic silicon carbide reveals a continuing lattice expansion with increased dosages of fast-neturon flux. The irradiation of these materials was carried out by means of a survey experiment in position C-39 of the LITR. During a nine-week period of bombardment, the LITR power totaled over 1.5×10^6 kw-hr and the lattice constants of both materials were still increasing. A qualitative plot of this relationship is shown in Fig. 28. These fragmentary data only serve to show that a detailed quantitative analysis of the phenomena is needed to furnish additional information concerning the process of irradiation damage. The investigation of these and other materials is being continued.

The irradiation of quartz under the same conditions as described for diamond and silicon carbide resulted in a lattice expansion comparable to that in diamond. The lattice strain imposed by this expansion is of considerable magnitude as seen in x-ray reflections at large Bragg angles, which are "smeared out" to near background level. Single-crystal studies are being undertaken by the X-ray Section.

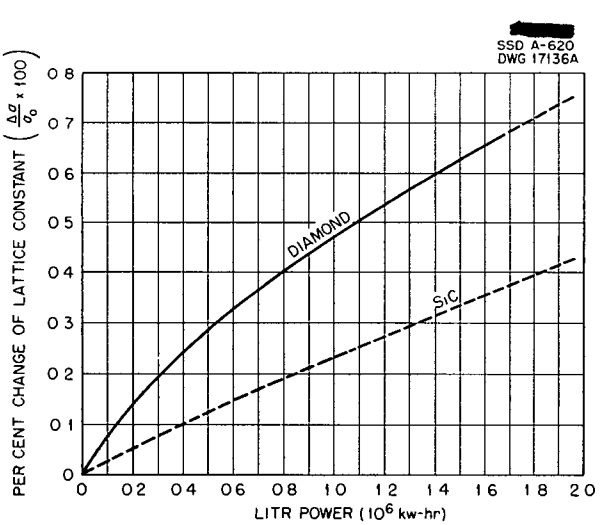


Fig. 28. Expansion of Material Under Fast-Neutron Bombardment.

MATERIALS IRRADIATION

R. H. Kernohan

Samples of two types of tool steels mentioned in the previous quarterly report[1] have been canned in four slugs and sent to Hanford for irradiation (Hanford Special Request No. 166).

The Hanford containers are being revised for request No. 167 because of insufficient wall thickness in the cap. The samples of ceramics for this irradiation that are being prepared by J. R. Johnson of the Metallurgy Division are nearly complete.

Samples of thorium metal prepared by R. E. Adams of the Metallurgy Division were tested, canned, and placed in hole C-59A of the LITR for a six-month exposure. Unfortunately, after two months of irradiation, one of the two containers that held the tensile test specimens was badly bent in handling by operations personnel. The other container, holding two rods and several hardness disks, is still in the LITR. Preirradiation physical measurements on the thorium specimens gave the following average results

Modulus of elasticity	10.0×10^6 psi
Electrical resistivity	18.81 μohm-cm
Density	11.647 g/cc
Hardness	60.6 DPH

The second irradiation of Fe_2O_3 powder in the water-cooled hole of the ORNL graphite reactor was completed. Previous tests reported in ORNL-1359 had shown no changes in antiferromagnetism. E. O. Wollan of the Physics Division reports that the second irradiation of the Fe_2O_3 powder also showed no significant changes, and this experiment will be discontinued.

During the past quarter, an attempt was made to compile some of the initial data on several of the remotely controlled devices that measured physical properties in the hot cells. The purpose of these data summaries was to evaluate the reliability and consistency of the results obtained on the various pieces of apparatus.

[1] R. H. Kernohan, *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

Part II

UNCLASSIFIED PROJECTS

CONTENTS

	Page
PART II UNCLASSIFIED PROJECTS	39
SUMMARY	43
SOLID STATE REACTIONS	44
Low-Temperature Bombardment of Germanium	44
Stable Energy Levels in Bombarded Germanium	44
Magnetic Susceptibility of Germanium	45
Annealing of Potassium Chloride *F* Centers	47
Radiation Effects in Ionic Crystals	48
ENGINEERING PROPERTIES	49
Radiation Stability of Plastics and Elastomers	49
SPECIAL PROJECTS	52
Materials Irradiation	52

SUMMARY

SOLID STATE REACTIONS

Low-Temperature Bombardment of Germanium. Exploratory low-temperature irradiations of single-crystal germanium indicate that fast-neutron bombardment produces a large amount of lattice disorder that is stable at temperatures below –78°C and readily anneals out at temperatures well below room temperature. The electronic energy levels associated with this thermally unstable disorder may be quite different from those that are stable at room temperature. Data concerning the nature of the energy levels associated with the low-temperature disorder and the annealing kinetics of the disorder are being analyzed.

A vertical liquid-nitrogen Dewar will be installed in hole 10 of the ORNL graphite reactor to permit the experiments to be extended to a lower temperature than is available in hole 52.

Stable Energy Levels in Bombarded Germanium. Recent low-temperature data obtained in the reactor cryostat indicate that thermally unstable disorder is retained by the germanium lattice at –78°C. This unstable disorder appears to have energy levels associated with it that are somewhat different from those stable at ambient temperature.

Studies of Hall coefficient and resistivity will be made over the entire range from liquid-nitrogen to room temperature. Preliminary data show that there is a continuous decrease in both resistivity and Hall coefficient with decrease in temperature. Previous predictions, based on initial slope experiments at –78°C, were that at temperatures below –70°C there would be a decrease in hole concentration with a corresponding increase in Hall coefficient.

Magnetic Susceptibility of Germanium. The liquid-nitrogen cryostat and iron bell-jar system has been fabricated and mounted over the ADL magnet. One filling of the cryostat will maintain the sample at –190°C for 15 hours. It has been necessary to add extra magnet shields around the magnetically sensitive components of the balance. The sensitivity of the balance is about 0.005 μamp, which is equivalent to about 0.0002 dyne.

Development is in progress of a field control system that is expected to hold the magnet current constant to better than 0.1%. In addition, the Hall voltage produced by a germanium crystal will be used to indicate field fluctuations. The fluctuations will be corrected by a variable current in the trimming coils of the magnet.

Annealing of Potassium Chloride *F* Centers. The *F* centers are electrons trapped in negative ion vacancies and *V* centers are holes in positive ion vacancies. Ionic conduction in potassium chloride is by means of positive ion vacancies, therefore the investigation of the *V* band would have a direct bearing on conductivity experiments.

The study of the *V* centers awaits the delivery of a Hanovia hydrogen lamp In the meantime, an investigation is being made into the behavior of *F* centers in the visible spectrum. Absorption spectra were measured at various times for a gamma-irradiated potassium chloride crystal that had been annealed successively at 120 and 150°C.

There appear to be two distinct energy bands in the bleaching of the *F* center. There also appears to be a saturation point in the low-temperature annealing so that complete bleaching does not occur.

An anneal was carried out at 200°C in an effort to eliminate the second band. Kinetic analysis of one run does not show either a simple first- or second-order process.

Radiation Effects in Ionic Crystals. New apparatus is being developed for measuring very low conductivities in solids. Evacuation of the sample region eliminates ionization produced by radioactive samples in a helium atmosphere. A vibrating-reed electrometer and a resistance-selector switch permit measurements of sample resistances over a range from 10^{-9} to 10^{-16} ohm.

Several annealing experiments have been completed with the improved apparatus, but the results have not been evaluated.

ENGINEERING PROPERTIES

Radiation Stability of Plastics and Elastomers. Formvar, Butacite, Pliovic, Geon 8600, Royalite, and Pliotuf were irradiated, and studies were made of the effects on tensile strength, elastic modulus, elongation, and hardness. Pliovic and Geon soften and form blisters filled with HCl. Royalite and Pliotuf exhibit excellent resistance to radiation.

Orlon, Dacron, and Nylon, in the form of filter cloths, were tested before and after irradiation. Nylon could be bent around a $\frac{1}{8}$-in. radius without

breaking, both Dacron and Orlon were hardened and embrittled beyond use. A special heat-treated Orlon fabric retained its flexibility and strength after an exposure of 4×10^{18} nvt.

Comparisons were made of the radiation effects produced in a number of elastomers by a Co^{60} source with effects produced by the ORNL graphite reactor for about equivalent exposures. The dosage required to produce a given change is roughly the same for the reactor as for Co^{60} at the same level of intensity. Cobalt-60 sources may therefore be utilized more frequently in future studies of organic materials.

SOLID STATE REACTIONS

LOW-TEMPERATURE BOMBARDMENT OF GERMANIUM

J. W. Cleland J. C. Pigg
J. H. Crawford, Jr. E. S. Schwartz

In the last quarterly report,[1] exploratory low-temperature experiments on single-crystal germanium were discussed in detail. These experiments indicate that fast-neutron bombardment produces a considerable amount of lattice disorder that is stable at temperatures below –78°C and readily anneals out at temperatures well below room temperature. In addition, these preliminary experiments seem to indicate that the electronic energy levels (hole and electron traps) associated with this thermally unstable disorder may be quite different from those that are stable at room temperature.

Since these early experiments, a number of single-crystal germanium samples (both *N*- and *P*-type), covering a wide range of original carrier concentrations, have been exposed under a variety of temperature conditions in the hole 52 cryostat of the ORNL graphite reactor. Large amounts of data, which concern both the nature of the energy levels associated with the low-temperature disorder and the annealing kinetics of this disorder, have been accumulated. At the present time, these data are being analyzed and correlated. The planning of further low-temperature bombardment experiments on germanium awaits the completion of this analysis.

It should be pointed out that similar low-temperature radiation effects have been observed by J. H. Forster[2] at Purdue University in connection with cyclotron bombardment experiments. Besides the disorder that is stable below –78°C, Forster has observed that deuteron bombardment produces another class of disorder that is stable at temperatures below about –150°C, a temperature range below the reach of the hole 52 cryostat. This very-low-temperature disorder appears to have electronic energy levels associated with it that are quite different from those associated with the more stable disorder. Although the temperatures used by Forster are beyond the range of existing in-pile low-temperature facilities, a metal liquid-nitrogen Dewar is now being constructed that will be installed in vertical hole 10 of the ORNL graphite reactor. This new facility will greatly extend the temperature range for in-pile experiments, and it will be possible to determine whether the very-low-temperature radiation effects observed by Forster with deuterons can be produced by fast-neutron bombardment.

STABLE ENERGY LEVELS IN BOMBARDED GERMANIUM

J. W. Cleland J. H. Crawford, Jr.

Although it has been conclusively demonstrated that both electron traps, which may act as acceptors, and hole traps are introduced below the middle of the forbidden-energy band of germanium

[1] *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

[2] J. H. Forster, private communication.

by fast-neutron bombardment,[3] the precise distribution of these states in energy is unknown. Previously, attempts have been made to determine the positions of these levels by analyzing conductivity vs. bombardment curves of *P*-type germanium irradiated at –78°C.[4] These attempts involved the fitting of both initial rate of change of hole concentration vs. initial hole concentration curves and conductivity vs. bombardment curves on the basis of appropriate energy-level models. Discrepancies between the fitting of these two types of curves were encountered. At that time the discrepancies were attributed to inadequacies in the models employed. However, the more recent low-temperature data obtained in the reactor cryostat indicate that the primary reason for these inconsistencies is due to the fact that, at –78°C, thermally unstable disorder is retained in some degree by the germanium lattice. This unstable disorder appears to have an appreciable annealing rate at dry-ice temperature and, in addition, appears to have associated with it energy levels that are somewhat different from those stable at ambient temperature. Consequently, all conclusions drawn from the initial slopes of conductivity vs. bombardment curves at –78°C, since they do not consider the possibility of annealing, must be discounted. At temperatures above those at which the low-temperature disorder is stable, this method is applicable in principle. However, at these temperatures the range of initial carrier concentrations required for the use of this approach lies so high (10^{16} to 10^{19} cm^{-3}) that bombardment times required for a measurable change in conductivity are prohibitively long. Consequently, a different approach is currently being used for investigating the locations of bombardment-produced energy levels.

Techniques have been developed for short-period reactor irradiations of a germanium sample in a cadmium shield with appropriate leads attached. After irradiation, Hall coefficient and resistivity measurements can be made as a function of temperature over the range from liquid nitrogen to room temperature. A 24-hr waiting period for radioactive decay is required between the time of the exposure and the measurements. A complete record of resistivity and Hall coefficient as a function of temperature is thus obtained at desired intervals during the bombardment period. Not only will it be possible to analyze individual curves at any stage of irradiation, but conductivity vs. bombardment curves can be constructed for any temperature of interest. In addition, this experiment makes possible the examination of the effect of fast-neutron bombardment on the mobility of charge carriers.

Preliminary Hall coefficient and resistivity curves are shown in Fig. 29 as a function of temperature taken during such an irradiation experiment. It is interesting to note that throughout the experiment there is an increase in hole concentration (a decrease in Hall coefficient) and a decrease in resistivity over the entire temperature range. This behavior is quite different from that expected on the basis of the –78°C initial slope experiments, since these would predict a decrease in hole concentration with a corresponding increase in the Hall coefficient at temperatures below about –70°C.

MAGNETIC SUSCEPTIBILITY OF GERMANIUM

D. K. Stevens

The construction of the liquid-nitrogen cryostat and the iron bell-jar system has been completed, and the units have been mounted over the ADL magnet. Testing and calibration of the assembled components is currently under way. When a 0.005-cm-thick copper foil is used to thermally connect the sample tube with the refrigerant vessel, there is a heat leak of 0.1 cal/sec from the outside into the sample region, with the result that –190°C is the minimum temperature obtainable. Preliminary tests indicate that one filling of the cryostat with liquid nitrogen will maintain the sample region at the minimum temperature for 15 hours. Provision for pumping on the refrigerant enables the attainment of lower temperatures. With liquid nitrogen as the refrigerant, the temperature of the sample tube may be raised to –163°C with a 2-watt heater input, –136°C with 4.7 watts, –99°C with 8.4 watts, and –58°C with approximately 14 watts. These operational characteristics appear to be satisfactory for the intended purpose, and thermocouple calibration is to follow.

The iron bell-jar system has been found to provide insufficient magnetic shielding for the

(3) J. W. Cleland, J. H. Crawford, K. Lark-Horovitz, J. C. Pigg, and F. W. Young, *Phys Rev* 84, 861 (1951).

(4) J. W. Cleland, J. H. Crawford, Jr., and J. C. Pigg, *Solid State Quar Prog Rep May 10, 1952*, ORNL-1301, p. 47.

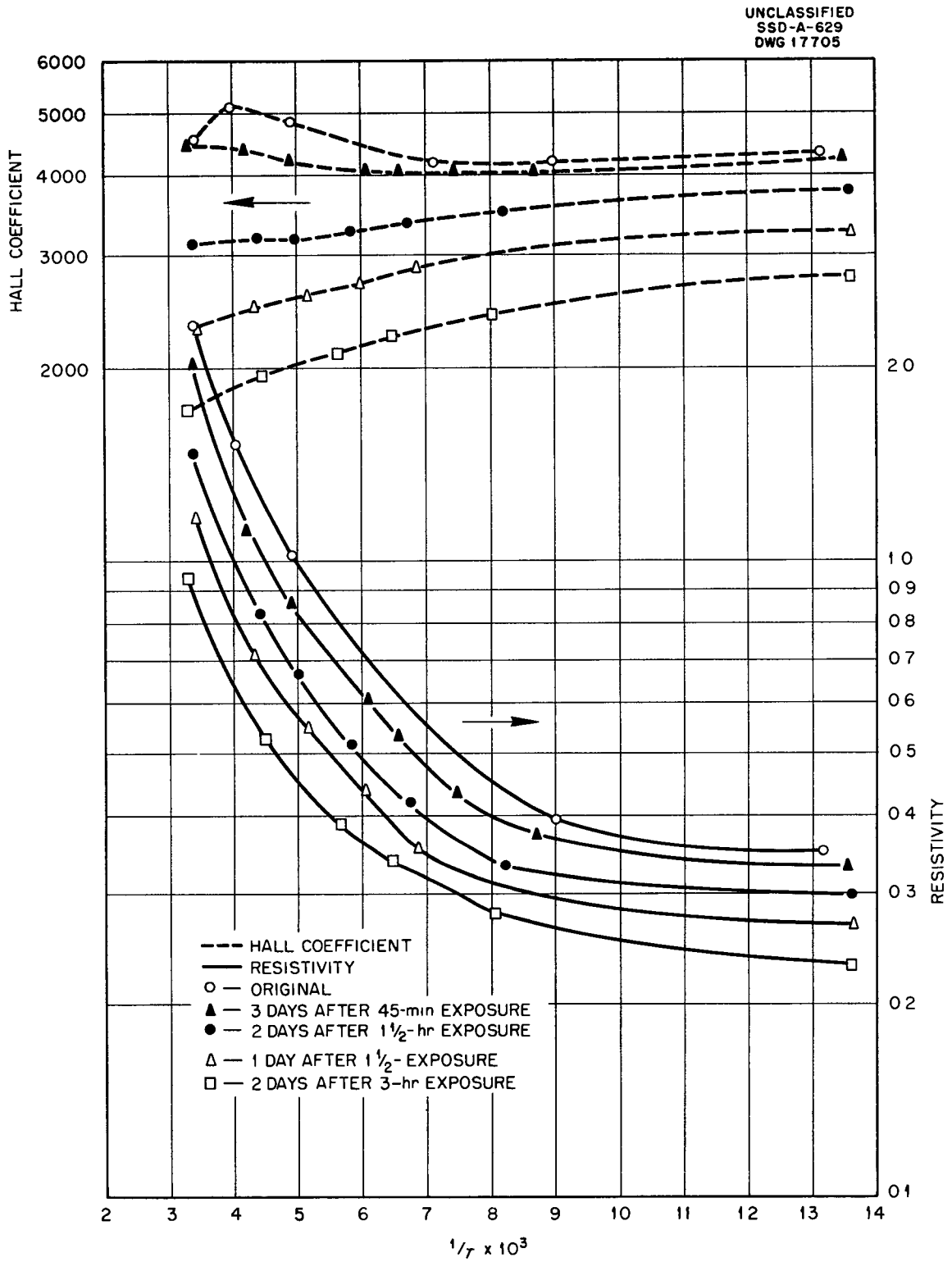


Fig. 29. Hall Coefficient and Resistivity vs. Reciprocal Temperature of *P*-type Germanium During Bombardment.

balance even though the external field is less than 10 gauss when the magnet is operating at 22 kilogauss. Nested shields of transformer iron around the magnetically sensitive components of the balance are being used, in addition to the iron tank, in the attempt to magnetically isolate the balance from the electromagnet. Fair, although not complete, success has been obtained. It has also been found necessary to replace the angle-iron mounts that support the iron vacuum system on the magnet with brass mounts in order to reduce conduction of magnetic fields to the base plate. Some difficulty was encountered in bracing this plate so that its position remained unchanged with state of excitation of the magnet. These problems have been of extraordinary importance in this instance because of the extreme sensitivity of the balance to position and magnetic field.

The sensitivity of the balance has been determined roughly as 28 μa/dyne with the limit of detectability being approximately 0.005 μa.

The vibration-isolation mounting system for the balance appears to be satisfactory, although it is still necessary to replace the coolant pump with one of smaller capacity. It was found that the present pumping system is powerful enough to cause the magnet to shift the magnet position when the pump is turned on. A replacement pump has been acquired but has not been installed. By bypassing the pressure switches, it is possible to operate the magnet for short periods of time with the pump off without excessive heating of the yokes and pole pieces.

A field control system is currently being dedeloped by J. C Pigg of this division A resistance-capacitance network is used to feed fluctuations of the generator output into one of the field coils of the amplidyne that supplies the field coils of the generator. Small fluctuations are thereby amplified and corrected at the source. It is expected that it will be possible to hold the magnet current constant to better than 0.2% by this method. The Hall voltage produced by a germanium crystal will then be used to indicate field fluctuations. The fluctuations will be corrected by a variable current in the trimming coils of the magnet. With this system, it should be possible to reset the magnet to any given field value without going through a tedious program of cycling the current, as is usually required because of hysteresis effect when the current alone is used to reproduce given fields. The range of the trimming coils is approximately 500 gauss when the magnet is operating at 25 kilogauss.

ANNEALING OF POTASSIUM CHLORIDE *F* CENTERS

D. Binder W. J. Sturm

According to the present ideas on color centers in alkali halide crystals,[5] *F* centers are electrons trapped in negative ion vacancies, and *V* centers are holes in positive ion vacancies. The annealing kinetics of the corresponding absorption bands are therefore of direct interest in the fundamental problem of the motion of vacancies in a crystal lattice. Because ionic conduction in potassium chloride is by means of positive ion vacancies, the investigation of the *V* band, stable at room temperature, would have direct bearing on the present conductivity experiments. Precisely how the hole attached to the vacancy will behave, and which is the more important in annealing, is a complicating feature and of interest in itself. An order was placed for a Hanovia hydrogen lamp for use in the study of the *V* band, and an investigation was initiated into the annealing of the *F* band of potassium chloride in the visible spectrum around 5600 Å.

A gamma-irradiated potassium chloride crystal was annealed successively at 120 and 150°C, and the absorption spectra at various times, *t*, are shown in Figs. 30 and 31 as measured in a Perkin-Elmer recording spectrometer. Here *D* is the optical density (log I_0/I) and *WD* is an arbitrary index of wavelength on the wavelength-drum drive of the spectrometer. The index decreases with increasing wavelength. It is interesting to note that Figs. 30 and 31 are successive chapters in the bleaching of the same crystal. In Fig. 30 at 120°C, the bleaching of the *F* center is the most striking characteristic, with some indication of the appearance, under equilibrium conditions, of a second band at longer wavelength, in Fig. 31 the temperature of the crystal was increased to 150°C, and the second (probably *F'*) center begins to disappear, approaching a second equilibrium density at longer times.

Two features are apparent from the data another band appears above *WD* = 1.1 (about 6400 Å), and there is a saturation in low-temperature annealing

[5] F. Seitz, *Rev Mod Phys* 18, 384 (1946)

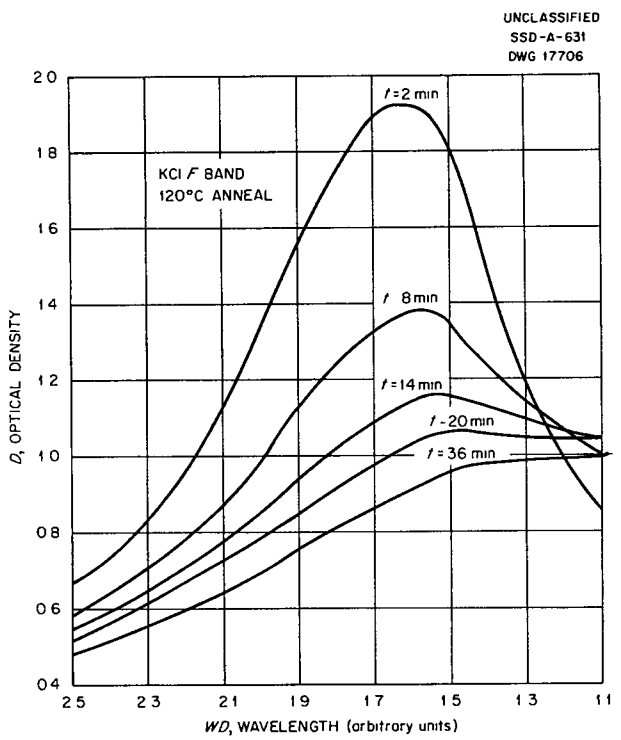


Fig. 30. Absorption Spectra for a Gamma-Irradiated Potassium Chloride Crystal after a 120°C Anneal.

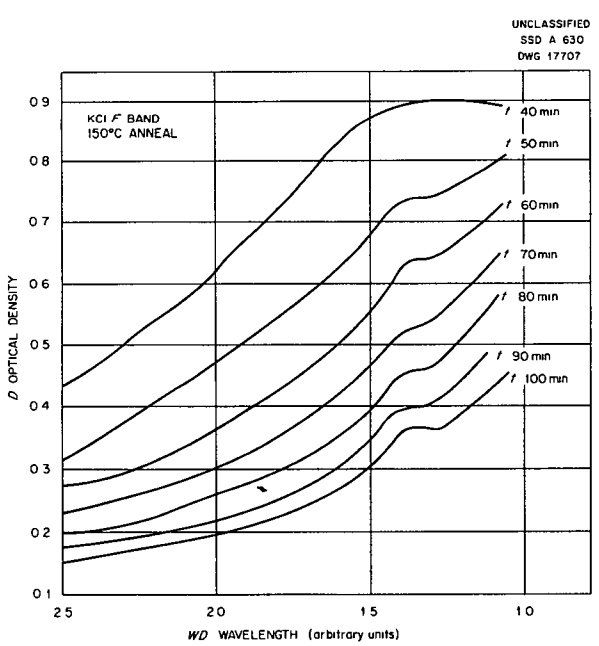


Fig. 31. Absorption Spectra for a Gamma-Irradiated Potassium Chloride Crystal after a 150°C Anneal.

(more apparent at 120°C), short of a complete bleaching. This is more graphically shown by plotting an individual wavelength. To examine the band at higher wavelengths (probably the F' band), requires either a reduction in the noise, coming mostly from the 13-cycle chopper amplifier, or a photomultiplier with a peak sensitivity much beyond that of the 1P21 detector at 4000 Å. The 5819 tube has a peak sensitivity at 5000 Å, but a much smaller absolute sensitivity at a voltage that does not saturate the signal.

The saturation of annealing short of complete bleaching indicates a recovery process, possibly the reaction $F'F$. In an effort to eliminate the complicating F' band and the recovery process, a 200°C anneal was carried out. The pertinent time interval was 6 min, so that only one wavelength could be followed thoroughly, and the half-maximum point at the low wavelength side of the band was chosen. This is both far away from the F' band and a point that does not vary with temperature in additively colored crystals. A kinetic analysis of one run does not show either a simple first- or second-order process, but further checks will be made.

RADIATION EFFECTS IN IONIC CRYSTALS

C. M. Nelson

Activity in the field of radiation effect in ionic crystals has been confined chiefly to the development of a new apparatus for the measurement of very low conductivities in solids. The improved apparatus may be evacuated in the region surrounding the sample and thereby eliminate spurious background current resulting from ionization produced by the radioactivity of the crystal in the helium atmosphere of the older apparatus. A vibrating-reed electrometer (Applied Physics Corp., model 30) is used to measure the very small currents produced by a 12-volt dry cell. A special electrometer turret switch (Applied Physics Corp., model 3097) was also secured. The switch had resistances of 1.0×10^8, 1.0×10^{10}, and 1.0×10^{12} ohms. With the ranges provided on the electrometer, a wide range of sample resistance can be covered (10^{-9} to 10^{-16} ohm). Care has been taken in construction of the apparatus to make sure that insulation with as high resistance as possible was used in the different components. When the apparatus was assembled, experiments demonstrated

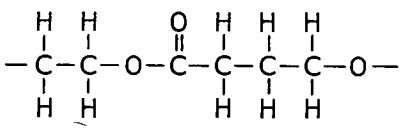
that more shielding was necessary, even though shielded cables and the usual precautions were used. Erratic readings and capacitative effects were observed. These effects were largely overcome by enclosing the entire apparatus in a metal cabinet. The advice and suggestions of J. C. Pigg in solving the shielding problems are gratefully acknowledged. Several annealing experiments have been completed with the improved apparatus, but the results have not been evaluated.

ENGINEERING PROPERTIES

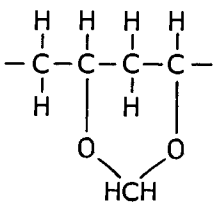
RADIATION STABILITY OF PLASTICS AND ELASTOMERS

C. D. Bopp R. L. Towns
O. Sisman W. K. Kirkland

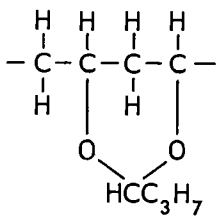
The materials recently examined are listed in Table 4. Polyvinyl formal, like polyethylene, eventually becomes embrittled. Unlike polyethylene, however, it initially increases in elongation and flexibility. Butacite also becomes more flexible, although it decreases in elongation. Materials previously reported[1] that show similar behavior are allyl diglycol carbonate polymer, Vulkollan, and Hycar PA. The structural formulas of these materials all contain $-C-O-C$, either as part of the main chain or bridging the main chain



Vulkollan



Polyvinyl formal (Formvar)



Polyvinyl butyral (Butacite)

Like Saran, a polyvinyl chloride plastic previously examined, Pliovic softens and blisters. The blisters are filled with HCl. Geon, a very flexible polyvinyl chloride composition, shows similar behavior.

The excellent radiation resistance of polystyrene raises the question of whether resistance will be imparted to mixtures or copolymers that contain styrene but have higher impact strength and flexibility than unmodified polystyrene. Royalite and Pliotuf are butadiene-styrene copolymers. Both are hardened, and at no period of exposure is their elongation better than polyethylene (a normally soft material with an aliphatic structure) with equivalent exposure, however, the amount of gas evolved during irradiation of Royalite is about one-tenth the amount from polyethylene.

Orlon and Dacron polymers are being tested in the form of filter cloths. After an exposure of 8×10^{18} nvt, Nylon filter cloth may be bent around a $\frac{1}{8}$-in. radius without breaking. Both Dacron and Orlon cloths, which were of similar weave to the Nylon, were so hardened and embrittled that they broke when an attempt was made to bend them. The Nylon and Dacron cloths showed little dimensional change. The Orlon cloth was gathered up and shrunken so that it was about two-thirds the initial size. The behavior of rigid Nylon sheet has already been described.[2] A special type of heat-treated Orlon fabric shows no change either in flexibility or tensile strength after an exposure of 4×10^{18} nvt. The material was obtained from Johns-Manville and resembles charred cloth, but unlike other kinds of charred cloth, it shows considerable strength. One type of weave shows a breaking load of about 40 lb for a 1-in.-wide strip of fabric.

Similar results are to be expected from fast-neutron and gamma radiation for organic materials that are damaged by ionizing radiation. Fast neutrons eject protons from hydrogen atoms, and the protons cause ionization and excitation of

[1] O. Sisman and C. D. Bopp, *Physical Properties of Irradiated Plastics*, ORNL-928 (June 29, 1951), O. Sisman, C. D. Bopp, W. K. Kirkland, and R. L. Towns, *Physics of Solids Institute Quar Prog Rep July 31, 1951*, ORNL-1128, p. 41.

[2] Sisman and Bopp, op cit, ORNL-928.

TABLE 4. PROPERTIES OF IRRADIATED PLASTICS

MATERIAL	EXPOSURE (10^{18} nvt)	TENSILE STRENGTH (psi)	ELASTIC MODULUS (psi $\times 10^5$)	ELONGATION (%)	ROCKWELL HARDNESS	
					(*a* scale)	(*R* scale)
Formvar (polyvinyl formal), 0.010-in. sheet	0	8000	4.2	2		
	0.8	5000	3.0	2.5		
	2.2	3000	1.4	30		
	5.6	4000	1.6	4		
	20.0	3300		<0.2		
Butacite (polyvinyl butyral), 0.015-in. sheet	0	1800		200		
	0.59	200		70		
	4.9	50		<0.2		
Pliovic (internally plasticized polyvinyl chloride), 0.090-in. sheet	0	7200	4.3	100	93	109
	0 55	6300	3 5	100	10	60
	1.1	6100	3.3	9	–5	40
	2 2	6300	3.5	5	63	85
Geon 8600 (polyvinyl chloride composition), 0 072-in. sheet	0	2200		280		
	2.6	200		20		
	4.9	2500		<2		
Royalite (styrene-butadiene copolymer), 0.060-in. sheet	0	3700	2.5	9	71	92
	0.25	4600	2.6	40	74	93
	0 76	6400	3.7	6	84	102
	2 2	7200	4.5	2	105	118
	5 6	5400	5.5	1	95 to 110	110 to 120
Pliotuf (styrene-butadiene copolymer and rubber mixture), 0.080-in. sheet	0	4800	2.8	4	81	88
	0.55	5000	3.0	1.5-6	84	94
	1.1	6000	3.1	6	90	99
	2.2	4000	4.0	1	99	109

susceptible molecules in their path in a manner similar to the secondary electrons produced by gamma radiation. Previously,[3] experiments were conducted with neutron-free gamma radiation from Au^{198} and with reactor radiation in which the gamma radiation was increased over that normally present with cadmium. To study the effect of the level of intensity more exactly, irradiations were made at various distances from a Co^{60} source in the ORNL canal. In Table 5, levels of intensity of 1.4×10^6 and 2.0×10^5 rep/hr are compared with reactor radiation (2×10^6 rep/hr for materials of about the hydrogen content of compounded elastomers) at roughly similar dosages in rep. The changes produced in elastomers were chosen for study because they are relatively sensitive and permit study at low intensity. The property that is most sensitive for elastomers is elongation.

On the basis of energy absorbed from the Co^{60} source, the low level of intensity is somewhat less effective in producing change than the high level of intensity, however, the difference is relatively small. Although results are not identical, the dosage of reactor radiation required to produce a given change is roughly the same as the dosage of Co^{60} radiation required to produce an equivalent change for the levels of intensity studied. The

[3] Sisman and Bopp, *op cit*, ORNL-928, C. D. Bopp, O. Sisman, R. L. Towns, and W. K. Kirkland, *Solid State Quar Prog Rep Aug 10, 1952*, ORNL-1359 (in press).

Co^{60} dosages were measured with an ionization chamber, the reactor exposure value for rep was calculated. Previous work with an Au^{198} source at a level of intensity of 1.5×10^4 rep/hr indicated that gamma energy absorbed at this level of intensity is about three times as effective as reactor radiation.[4] To check this result, a Co^{60} irradiation also is being conducted at this low level of intensity. In addition, the ion chamber used to measure the gold source will be checked against the ion chamber used to measure the cobalt source. The Co^{60} and reactor exposures give approximately the same results. This indicates the use of Co^{60} in future studies on organic materials because there is less limitation in specimen size and no problem as to induced radiation either in the specimen or in auxiliary equipment. At present, however, the Co^{60} facilities are very limited.

Additional specimens are being irradiated for x-ray diffraction study. Several reactor-irradiated rubbers proved too radioactive for study at the present time. It is planned to irradiate these materials with Co^{60}.

(4) Bopp, Sisman, Towns, and Kirkland, *op cit*, ORNL-1359 (in press).

TABLE 5. COMPARISON OF PHYSICAL PROPERTY CHANGES PRODUCED IN ELASTOMERS BY A Co^{60} SOURCE WITH CHANGES PRODUCED BY THE ORNL GRAPHITE REACTOR

ELASTOMER	RADIATION EXPOSURE	TENSILE STRENGTH (psi)	ELONGATION (%)	SHORE DUROMETER HARDNESS	SHORE ELASTICITY
Natural rubber	(a)	2900	430	68	100
32A43	(b)	2600	370	72	80
	(c)	2300	280	72	70
	(d)	2600	360	72	70
Neoprene W	(a)	2800	460	87	75
32A44	(b)	1700	130	88	60
	(c)	1300	50	85	40
	(d)	1800	80	92	
GR-S 50	(a)	1700	240	70	80
32A47	(b)	1550	140	77	80
	(c)	1050	50	83	50
	(d)	1200	80	82	70
Thiokol ST	(a)	750	130	75	40
3000 ST	(b)	770	105	78	40
	(c)	580	95	76	30
	(d)	730	105	74	30
Silastic 7-170	(a)	430	90	60	30
	(b)	490	35	76	20
	(c)	510	20	83	10
	(d)	550	30	81	15

(a) No irradiation

(b) Exposed in the ORNL graphite reactor for 0.07×10^{18} nvt (for materials of about this hydrogen content, the level of intensity of ionizing radiation is 2×10^6 rep/hr and the dosage is 5×10^7 rep).

(c) Exposed to a Co^{60} source at a level of intensity of 1.4×10^6 rep/hr for a dosage of 6×10^7 rep

(d) Exposed to a Co^{60} source at 2.0×10^5 rep/hr for 8×10^7 rep