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METALLURGY RESEARCH SUB-SECTION
JANUARY, FEBRUARY, MARCH
1956**

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METALLURGY RESEARCH SUB-SECTION
JANUARY, FEBRUARY, MARCH, 1956

Written by the members of the Metallurgy Research Sub-Section

S. H. Bush - Physical Metallurgy
J. J. Cadwell - Pile Metallurgy
L. D. Turner - Radiometallurgy
L. A. Hartcorn - Metallography Laboratory
W. R. Smith - Corrosion and Welding
O. J. Wick - Plutonium Metallurgy

April 17, 1956

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9. HW-39495 - Quarterly Progress Report - Metallurgy Research Sub-Section - July-Sept., 1955 - November 1, 1955.
10. HW-40858 - Quarterly Progress Report - Metallurgy Research Sub-Section - Oct.-Dec., 1955 - February 1, 1956.

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SUMMARY

PHYSICAL METALLURGY

Mechanical and Physical Properties of Fissionable Materials

Tensile specimens of natural uranium irradiated to 0.4 , 0.85 , and 1.8×10^{20} nvt have been annealed at 400 C for times as long as 100 hours and at 600 , 700 , and 800 C in the case of the longest irradiation, again for times to 100 hours. These specimens will be tested at room temperature in the next quarter.

Electrical resistivity of uranium irradiated to 0.4×10^{20} nvt has been determined over the range 25 to 100 C. Resistivity was essentially the same as that in the unirradiated control specimens.

Metallographic specimens of U-1.5 a/o-Si, U-0.35 a/o Cr, U-23 a/o Si, U-0.5 a/o Ti, U-12 a/o Mo, and Zr-480 ppm hydrogen were inserted in NaK filled containers, then heated to 400 , 600 , and 770 C for four weeks to see if there was a NaK-metal reaction. Some evidence of interaction between the nichrome separators and the uranium alloy was observed.

Dimensional stability specimens of depleted uranium containing 20, 25, and 35 per cent cold work have been prepared for irradiation at the MTR.

The thermal conductivity of unirradiated, natural, beta-heat-treated uranium has been determined from 134 to 218 C. Values ranged from 0.0691 to 0.0713 cal/cm/sec/C. These results compare favorably with work of other investigators.

An X-ray investigation has been made of the pre- and post-irradiation preferred orientation of uranium specimens subject to varying degrees of dimensional instability. Definite shifts in rho values for (010), (110), and (100) planes and for (100)/(110) ratios were observed in the irradiated specimens. The shifts were very marked in the specimens exhibiting the greatest degrees of dimensional instability.

Uranium tensile specimens have been irradiated in an unperturbed flux of 2 to 3×10^{14} nv at the MTR for 15 days. This flux should result in temperatures in the beta-gamma region.

The behavior of copper and uranium rods fabricated by swaging spiral wound sheet about a solid mandrel has been investigated. Densities of 99.7 per cent that of swaged solid rod were obtained. Post-sintering thermal expansion values were essentially the same for composites and for solid rods. No evidence of grain growth across the sheet interfaces

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was observed even though it was not possible to rupture the bond between the uranium sheets.

Flow stress recovery of cold-worked dingot uranium is being studied as functions of temperature, annealing atmosphere, and rolling direction. Pronounced differences in the elongation were observed between transverse and longitudinal specimens after annealing in a static vacuum whereas much less pronounced changes were observed in a dynamic vacuum. These differences are attributed to contamination.

Reactor Structural Materials

Twenty-four Zircaloy-2 tensile specimens are currently being irradiated at the MTR to an integrated fast neutron (>1 Mev) flux of about 1.5×10^{20} nvt. Twelve have been discharged, and the remaining twelve are scheduled for discharge in April.

Vacuum annealing of zirconium and Zircaloy-2 after preliminary oxidation, results in a pronounced acceleration of the reaction rate during subsequent oxidation. No such change is observed if vacuum annealing precedes oxidation. The cause of this phenomenon is being investigated further.

The epitaxial relationship between the grains of zirconium and zirconium oxide is currently being investigated. Oxide films appear to consist of grains similar in size to the underlying metal grains and bearing a definite orientation relationship to these metal grains. Preliminary results indicate the (111) plane in the oxide is aligned with the (0001) plane in the zirconium.

The kinetics of recrystallization and recovery of zirconium, Zircaloy-2, and Zircaloy-3 are being determined. Per cent cold work, temperature, time, and heat treat atmosphere have been selected as variables. Base anneal conditions have been selected; these consist of a vacuum anneal of one hour at 600 C.

Fundamental Studies

The microstructure of uranium has been examined over a range of magnification of three to 30,000 diameters using replicas and optical and electron microscopy. By combining these procedures, it is possible to observe the structure in the same field to the limits of resolution of both the optical and electron microscopes. The correlations obtained can be applied to irradiated uranium yielding valuable information.

Electron micrographs of uranium irradiated to 1.5×10^{20} nvt have been observed to 3500 diameters. These micrographs exhibit extensive twinning

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and, possibly, slip.

The diffusion of U-Zr couples is being investigated at 450 C utilizing hot pressing to facilitate bonding. Prebonding at 700 C followed by 450 C vacuum annealing is also under investigation. Diffusion in U/AlSi couples annealed 28 days at 255 C was observed to be about 0.003-inch with the uranium diffusing into the AlSi.

The crystallographic orientation of a deformed uranium specimen has been observed after vacuum cathodic etching. The grain studied had a (001) face exposed. Predominant twins were [130] with etch pits delineating [172] or [176] twins. A (100) kink boundary was also observed.

The diffusion coefficient of krypton gas in silver at 735 C was found to be $D_{735} = 4.3 \times 10^{-8} \text{ cm}^2/\text{sec.}$ compared to LeClaire and Rowe's value $D_{735} = 5.7 \times 10^{-9} \text{ cm}^2/\text{sec.}$ The radioanalysis technique consisting of irradiating the krypton gas then counting with a gamma spectrometer has been extended successfully to xenon. Radioxenon is more satisfactory than radiokrypton due to its longer half life. Additional diffusion experiments are under way with both krypton and xenon in silver and other metals.

Precision back reflection x-ray diffraction patterns on zirconium irradiated to 6.5×10^{18} and 6.5×10^{19} nvt revealed no change in lattice parameters in the low exposure specimen and only a slight shift at the high exposure.

A document, HW-42022, entitled, "Neutron Irradiation of Sapphire (Al_2O_3)" was released this quarter.

Equipment and Facilities

An evacuable hot stage unit containing a mechanism for loading micro specimens in tension, compression, or bend has been designed.

A prefocusing x-ray goniometer has been designed to investigate deformation mechanisms and to preselect any grain or grain area to permit specific structures to be examined.

PILE METALLURGY

Fuel Element Fabrication

Experiments with swaging as a method of fabrication of uranium oxide fuel elements have shown that swaging at 750 C can cause growth of large crystals in UO_2 powder. Experiments are continuing to determine a swaging schedule which will close the shrinkage cracks which arise during crystal growth.

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Clusters of fuel rods are being studied as an extended surface geometry which can be adapted for irradiation in horizontal process tubes. A four-rod cluster was made up which contained 54% of the uranium contained in a standard HAPO slug per foot of fuel element. Rupture of a defected four-rod cluster in a process tube of KER geometry containing 260 C water resulted in no appreciable flow reduction, whereas rupture of a standard Hanford slug led to rapid and severe flow blockage. 305 Pile tests of the proposed four-rod cluster indicated the cluster in the KER tube would generate about 1.02 times the power per foot of fuel element that a standard solid slug would generate in a standard KE tube. The test was made utilizing rods clad in stainless steel and the calculations assumed a Zircaloy-2 process tube. The spider supporting the rods is still under development.

Fuel Element Evaluation

An analysis of the thermal stresses and strains in solid fuel elements has been completed utilizing a time and temperature dependent model. In one case calculated, the fuel element was assumed to operate between 100 and 350 C and in a second, between 350 and 600 C. In both cases the calculations show that considerable stress relaxation occurs in the uranium during start-up. In the high temperature case the central portion of the slug achieves an essentially stress-free condition shortly after steady state conditions are reached.

Insulated fuel elements are being studied to determine the in-pile stability of uranium slugs operating at elevated temperatures. During the second experimental irradiation of insulated elements in the MTR completion of burnup analyses from the first experiments indicated power levels higher than anticipated. Calculations showed that melting should have occurred in these slugs. The three slugs have now been stripped and examined. Melting varied from complete closure of the axial core in one slug to partial closure in the second and no movement of metal in the third.

During the installation of facilities to control the storage and disposal of fission products from the GEH-4 facility in the MTR, a measurement of gamma heat generation in the facility was carried out with a thermocouple lead slug. After the reactor reached full power, calculations showed that gamma heating in a uranium slug would be 1 watt/gram.

An experimental study of samples simulating canned, unbonded uranium fuel elements is being carried out. The specimens are heated prior to exposure to water and can be heated during exposure. Specimens exposed to 285 C water are attacked in a rapid and non-uniform manner if the uranium temperature is held at or below 450 C. A specimen held at uranium temperatures of 720-750 C was not attacked by hydrogen, and the rate of formation of uranium oxide was reduced.

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Fuel Materials

Uranium-magnesium matrix fuel material is being studied for high burnup applications. In an experimental irradiation in the MTR, four 0.040-inch diameter specimens canned in Zircaloy-2 have been irradiated to 1000 MWD per ton of uranium, four to 500 MWD/T, two to 10,000 MWD/T, and two to 20,000 MWD/T. The specimens were dimensionally stable at all exposures. Bend tests of the samples gave identical curves, within experimental error, for the 1000, 5000, and 10,000 MWD/T samples. Ultimate load decreased slightly for the 20,000 MWD/T samples. Some loss of ductility occurred in the magnesium matrix, but this effect appeared to saturate at 0.1 percent burnup. Studies of uranium oxide as a reactor fuel material are being continued. A ceramic fabrication laboratory will commence operation during the next quarter.

Metal coalescence studies have been carried out to investigate the use of vibration to increase coalescence of metal during bomb reduction. In the reduction of cerium dioxide by calcium, use of a 60-cycle/second vibrator during the bomb reduction increased the yield of metal in button form by 33 to 67 percent.

RADIOMETALLURGY

Continued examination of the slugs adjacent to the ruptured slug in the H-loop has shown that extensive intergranular corrosion and some diffusion of the Al-Si into the aluminum can wall has taken place. However, even where the bare uranium is exposed to the process water, little or no evidence of uranium corrosion was seen. Metallographic samples from the slug upstream from the rupture have also indicated that temperatures in excess of the Beta transformation temperature were realized.

The Zirconium tube used in the H-loop was sampled for hardness testing and gas analyses. Preliminary results show no significant increases in hardness values but the hydrogen content, in the rupture area, appeared high enough to cause some embrittlement and an increase in hardness.

One slug, No. 34, from Run V, Bluenose, irradiation was found to have a symmetrical distribution of gamma fission products across its diameter and was prepared for additional sectioning to provide confirmatory chemical analyses.

Two slugs that were warped to the extent of .010" and 0.088" were examined to determine whether hardness, flux asymmetry and degree of warp could be observed. There also was no significant variation in hardness between the convex and concave side of the two slugs. Orientation values have not yet been ascertained.

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Four slugs were received to evaluate the current type of cap failures. Three of these slugs were not ruptured but showed exterior markings indicative of possible future failure. The slug measurements showed a general decrease in length and an increase in diameter except for the ruptured slug which had lengthened .029 inches.

Three slugs which had ruptured between the ribs have been received to determine the type of corrosion between the ribs and the rupture mechanism. Each slug has a 1/2" to 3/4" hole between the ribs 1" to 2" from the cap end. Wafers 1/2" thick were taken from each slug for detailed metallographic examination.

Gamma scintillation scanning of one of the solid Al-Si bonded slugs that was irradiated in the MTR showed flux asymmetry of 50 percent from side to side. No warp or other dimensional instability was associated with this highly asymmetrical flux distribution. Rockwell hardness values showed no unusual heating effects within the uranium. However, the hardness values of the cored AlSi bonded slug irradiated at the same time did show a noticeable variation in hardness that may be correlated with its flux asymmetry.

Metallographic examination of the failed, internally and externally cooled, nickel-plated, hot-pressed slug, which was irradiated in the MTR, has revealed considerable intergranular corrosion to the aluminum in the inner can wall. Gamma scintillation scanning of wafers from each of the three slugs tested shows a maximum flux difference of 19 percent at points equidistant from the wafer center but along different diameters.

Metallographic techniques were improved during this quarter by making X-ray and metallographic investigations of various grinding-polishing etching procedures on non-irradiated uranium. The metallographic cell has been renovated and functioned satisfactorily. However, a new project has been started that will increase the metallographic capacity.

A new X-ray sample holder and a sample transfer cask were acquired during this period. Design of a one-ton laboratory transfer cask was completed and purchase specifications prepared.

CORROSION AND WELDING

Samples of type 304-L and 347 stainless steel which had been subjected to a sensitizing heat treatment, and samples of A-55 titanium in the "as-received" condition were exposed to solutions of 10 w/o oxalic acid for time periods ranging from 0.5 to 100 hours at temperatures ranging from 25 C to 101 C. The results of these exposures indicate that 10 w/o oxalic acid at temperatures of 80 C to 100 C appears to be a satisfactory

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repetitive cleaning agent, from a corrosion viewpoint, for stainless steel process equipment where relatively large corrosion rates can be tolerated for short periods of time. Corrosion rates of the two stainless steels investigated ranged from 0.006 to 0.020 ipm; three to ten times the corrosion rates observed for these materials in boiling 65 w/o nitric acid. Oxalic acid in most concentrations at temperatures above 35 C does not appear to be a satisfactory repetitive cleaning agent for titanium process equipment, unless extremely large corrosion losses can be tolerated. Titanium exhibited corrosion rates on the order of 0.100 ipm in 10 w/o oxalic acid at 101 C; 500 times the rate exhibited in boiling 65 w/o nitric acid, and 0.5 w/o oxalic acid at 35 C exhibits a corrosion rate of 0.0001 ipm; almost equal to the rate observed in boiling 65 w/o nitric acid.

The expected serious corrosion problem in the Purex Acid fractionators has made the investigation of nitric acid corrosion inhibition highly desirable. Other investigators have reported significant reductions in corrosion by nitric acid using urea as an inhibitor, however, previous work performed by this laboratory did not confirm these results. Re-examination of the testing techniques used by this laboratory, and slight modifications of these techniques have resulted in agreement with other investigators. It appears that the addition of as little as 0.01 M urea to 65 w/o nitric acid may reduce the observed corrosion rate by as much as a factor of two to three.

In order to relate the corrosion rates derived by autoclave type experiments to the corrosion rates obtained using heat transfer equipment, accurate measurements of the heat transfer surface temperature and the bulk metal temperature of the heat transfer specimen were made. These measurements indicate that the surface temperature is 20 to 30 C less than the bulk metal temperature. A comparison of temperature corrected corrosion rates from the heat transfer units and corrosion rates from autoclave type tests reveals close agreement between the two methods.

The anticipated corrosion of selected candidate materials for heat transfer surfaces in the Purex waste acid concentrators has been evaluated. Specimens of type 312 stainless steel and grade A-55 titanium were tested as heat transfer surfaces in synthetic Purex waste acid concentrate. The corrosion rates of sensitized type 312 stainless steel (0.09 w/o C) ranged from 0.0012 ipm when the metal temperature was 115°C to 0.0030 ipm when the metal temperature was 165°C. The corrosion rates of annealed grade A-55 titanium were less than 0.0005 ipm, over the metal temperature range of 115 - 165°C. Similar experiments on sensitized type 312 stainless steel with the carbon content lowered to 0.03 w/o indicate the corrosion resistance of the low carbon material may be better by as much as a factor of two.

Five corrosion test heat exchangers were placed in the F-6, F-11, H-4, J-8 and K-4 concentrator overflow chambers in the Purex facility this month. These units are single tube calandria type heat exchangers

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consisting of a 2" schedule 40 stainless steel pipe shell, 3/4" schedule 40 stainless steel tube and supplementary steam inlet and condensate return lines. A sample rack was attached to the end of each unit to hold corrosion coupons for in service static tests. The test units will be operated in the overflow chambers under the same conditions as the process heat exchangers. These facilities will be used to obtain a better understanding of:

1. Where will the heat exchanger fail first, i. e., what is the weakest link in the concentrator design and construction?
2. How significant are the advantages of using type 304-L vs type 347 stainless steel construction materials and what are the differences in actual field service between types 308-L and 347 welding alloys?
3. Can service life expectancies be predicted on the basis of the data collected by our laboratory testing techniques, i. e., is there good correlation between laboratory data and field experience?

Remote Studs

Several type 414 stainless steel studs which were welded to vessel flanges and installed in the Purex plant failed by brittle fracture during activation of the Purex facility. As a result of these failures, replacement of all "critical" studs was initiated, utilizing studs fabricated from two lots of type 420 stainless steel. Immediate failure of several replacement studs initiated an investigation to determine the optimum material and heat treatment for this service. Torsion impact and tensile tests were performed on several candidate materials which had undergone various heat treatments. The results indicate that types 414 and 416 stainless steel, tempered at 500 - 600°F are the better materials for this application.

Mercury Switches

The integrity of the primary alarm circuit of HAPO reactors depends upon the reliability of the mercury switches which actuate the alarm circuit. After about two months' operation, some of the Hamlin mercury switches containing graphite pivots are becoming "sluggish" or "sticky" and some of the switches fail to reset after actuation. Twenty three mercury switches including five "sticky" switches have been disassembled and examined. The following conditions are apparent: 1) the presence of mercury between the graphite bearing and nickel journal, 2) particulate graphite suspended in this mercury, and 3) extensive deterioration of the inner surface of the graphite bearing. It is suggested that this problem can be solved by replacing the nickel journals with stainless steel journals.

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The technology of Zircalloy-II fabrication has not been developed to the extent that full length HAPO reactor process tubes containing ribs can be extruded. However, techniques have been developed which produce satisfactory butt welds for joining shorter lengths of this tubing. Experimental tubes for reactor evaluation have been joined and tested by radiographic examination. Although, the radiographs and visual examination indicate acceptable welds, the post-pile-exposure examination of the experimental tubes will serve as a better evaluation of this welding method.

Pipe Joints

The "GE" pipe joint configuration offers an advantage over conventional joints from the standpoint of integrity, inspectability, and fabrication requirements. To permit rapid and accurate joint preparation, form cutting tools have been developed. The two tools developed will prepare the "GE" joints on stainless steel and carbon steel pipe with wall thicknesses ranging from 3/32" to 3/4".

Roto-Arc

In support of the concerted effort at HAPO to develop automatic fuel element canning equipment, the "Roto-Arc" welding process, which is amenable to automation, has been developed and demonstrated. The "Roto-Arc" process utilizes a cylindrical electrode which, to date, has been fabricated from molybdenum. As an attempt to gain the advantages of tungsten as an electrode, a cylinder of sintered tungsten was fabricated into an electrode. This high density sintered material will not withstand the rapid thermal cycling necessary and fails in a brittle manner. A continued effort is being made to develop a suitable tungsten electrode.

In an effort to improve the safety of operation and the quality of welding at HAPO, a new type welding machine, the AC-DC rectifier welder, has been tested and evaluated. It is concluded that the AC-DC rectifier welder will effectively perform the same operations as both the transformer and motor generator type welders. This machine is economical to operate and several new safety features have been incorporated into the equipment. For these reasons, it is strongly recommended that all replacement and/or new welders obtained at HAPO be of the AC-DC rectifier type.

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PHYSICAL METALLURGY - S. H. Bush

Mechanical and Physical Properties of Fissionable Materials

HAPO Irradiation - P. T. 105-3N - R. E. Hueschen

The testing of tensile specimens irradiated under Production Test 105-3N has been completed. All additional tensile testing will be done on specimens irradiated under P. T. 105-3N, Supplement A.

HAPO Irradiation - P. T. 105-3NA - R. E. Hueschen, W. S. Kelly, R. S. Kemper, and L. J. Chockie

All of the uranium specimens irradiated under P. T. 105-3N, Supplement A, to 0.4, 0.85, and 1.8×10^{20} nvt have been removed from their containers by Radiometallurgy personnel. In addition, ten tensile specimens have been vacuum annealed at various temperatures and times for room temperature testing as shown in Table 3.1.

TABLE 3.1

ANNEALING OF IRRADIATED TENSILE SPECIMENS (PT-105-3NA)

<u>Exposure</u>	<u>Number of Hours Annealed at Temperature</u>			
	<u>400 C</u>	<u>600 C</u>	<u>700 C</u>	<u>800 C</u>
0.4 $\times 10^{20}$ nvt	100			
0.85 $\times 10^{20}$ nvt	100			
1.8 $\times 10^{20}$ nvt	10, 25, 100	10, 25, 100	10	10

Electrical resistivity measurements were made on a uranium specimen irradiated to 0.4×10^{20} nvt (150 MWD/T). In comparison with pre-irradiation data, the resistivity of this specimen in the temperature range from 25 to 100C was unchanged after irradiation. Since the irradiation temperature of this specimen was approximately 350 C (660 F) little change in resistivity would be expected from vacancy-interstitial pairs. The total fission product concentration after this exposure is about 0.037 a/o, of which 0.03 a/o are metal atoms and 0.006 a/o are inert gas atoms. An increase in resistivity would be expected from these impurity atoms. It is felt that if the measurement had been made at a much lower temperature, instead of at and above room temperature, some increase in resistivity would have been noted. Microcracks would be expected to increase the electrical

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resistivity and this did not occur under the irradiation conditions. If dislocation loops are formed as postulated by Brinkman, (1) these should collapse at the exposure temperature which existed. From the Wiedemann-Franz relationship, it may be assumed that little change in thermal conductivity has occurred under the stated irradiation conditions.

Reaction of Uranium Alloys and Zirconium with NaK - R. G. Wheeler and L. F. Kendall

It was assumed that the reaction between NaK and uranium alloy tensile specimens exposed in the MTR was negligible. In order to confirm this assumption, metallographic specimens of U-1.5 a/o Si, U-0.35 a/o Cr, U-23 a/o Si, U-0.5 a/o Ti, U-12 w/o Mo, and Zr-480 ppm hydrogen were exposed to NaK for four weeks at three temperatures, 400, 600, and 770 C (750, 1112, and 1420 F). The metallographic constitution of identified areas was recorded prior to exposure for comparison with the same areas after exposure; however, the post exposure metallography is incomplete at this time. The specimens exposed at 770 C reacted locally with Nichrome fixtures in the can probably because of the formation of a low melting eutectic.

Irradiation of the specimens at the MTR was completed in January, but lack of casks has prevented shipment to HAPO.

Effect of Irradiation on Depleted Uranium Containing 0.03 w/o U-235 - R. E. Hueschen

It is known that neutron irradiation of normal uranium containing 0.7 w/o U-235 drastically affects its tensile properties. This program consists of obtaining the properties of irradiated depleted (0.03 w/o U-235) uranium. Depleted uranium rods have been cold swaged to approximately 20, 25, and 35 per cent reduction of area to provide dimensional stability specimens with various degrees of preferred orientation. These specimens will be canned in NaK with control specimens for irradiation in the Materials Testing Reactor.

Thermal Conductivity of Uranium - R. L. Hales

Thermal conductivity for one beta heat treated specimen of uranium has been determined from 134 C to 218 C. Experimental results ranged from 0.0691 cal/cm/sec/C at 134 C to 0.0713 cal/cm/sec/C at 218 C compared to the range of 0.067-0.0712 at 134 C and 0.0696-0.0753 at 218 C reported by British investigators. Further data has been delayed pending re-design of vacuum apparatus. Sample oxidation dictates that a static vacuum be maintained for the one week required for examination. Re-design will incorporate flexibility of specimen geometry and an increased temperature range.

(1) All footnotes are referenced at the end of this section.

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Physical Properties of Uranium Subjected to Electron Bombardment -
R. L. Hales

Evaluation of a portable vacuum liquid nitrogen cooled cryostat design is in progress. Such a design would allow low temperature bombardment and subsequent annealing studies.

Dimensional Stability of Uranium - W. V. Cummings and C. L. Boyd

The effect of preferred orientation on the dimensional stability of uranium during irradiation has been well demonstrated. In way of review, single crystals elongate in the $[010]$ direction, shorten in the $[100]$ direction, and remain essentially stable in the $[001]$ direction. Some experimenters who have irradiated nearly perfect crystals have assigned quantitative values to growth coefficients for the three crystal axes. The deformation of preferentially oriented polycrystalline material during irradiation occurs in the same crystallographic directions as in the single crystals, but the magnitude and type of deformation depends upon several structural factors. At the present time only qualitative correlations have been determined. Several differences in the behavior of the single crystal and polycrystalline material have been observed that are unexplained to date. For instance, growth rates more than twice as great as those measured in single crystals have been observed in highly oriented polycrystalline material.

A study of crystalline changes as a result of irradiation should indicate possible mechanisms of deformation in uranium. Several specimens, cut from irradiated material, have been examined by x-ray diffraction to determine preferred orientation changes. Since pole figures have been impossible to obtain, orientation values were taken from surfaces normal to the axes of the cylindrical parent material. The values are expressed as ρ (rho) which is defined as the ratio of the diffracted intensity from an (hkl) plane in the test material to the diffracted intensity from the same (hkl) plane in randomly oriented uranium. The pre-irradiation and post-irradiation data and also the dimensional changes of these specimens are given in Table 3.2. It is noted that for each type of oriented material, a trend toward a definite type of orientation change is developing. No attempt will be made to explain this phenomenon or predict the resulting behavior at this time. Other specimens are being studied in an effort to obtain more conclusive data for some types of oriented material. In addition, since these specimens received identical exposures of 150 MWD/T (4.4×10^{19} nvt), data as a function of exposure are also desirable before a final analysis is made.

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TABLE 3.2

DIMENSIONAL AND CRYSTALLOGRAPHIC ORIENTATION CHANGES AS A RESULT OF IRRADIATION

Sample #	$\bar{\rho}(010)^*$		$\rho(110)$		$\rho(100)$		$\rho(100)/\rho(110)$		Crystallographic Orientation Description		Length Change μ in/in/MWD/T
	Pre	Post	Pre	Post	Pre	Post	Pre	Post	Pre-Irradiation	Post-Irradiation	
A-1	1.4	1.2	3.8	7.9	6.2	4.1	1.6	0.52	(100) and (110)	(100) and (110)	- 85.6
A-3	1.1	1.1	6.4	7.7	7.0	3.7	1.1	0.48	planes normal	planes normal to	- 84.0
A-5	1.3	1.3	3.5	9.8	4.8	5.2	1.4	0.53	to rolling direc-	rolling direction	- 83.7
A-4	1.4	1.1	2.9	9.5	2.2	2.5	0.76	0.26	tion with (100) predominating.	with (110) pre-dominating.	
							$\rho[010]/\rho(110)$				
							Pre	Post			
B-1	2.4	2.8	2.0	0.86	0.70	0.93	1.2	3.3	(010) and (110)	(010) and (110)	+116.4
B-5	3.4	4.3	2.5	1.5	0.36	0.61	1.3	2.9	planes normal	planes normal to	+113.0
C-9	2.9	3.0	2.9	1.7	2.4	2.1	1.0	1.8	to rolling direc-	rolling direction	+114.0
C-6	4.1	6.3	2.4	1.0	0.41	0.29	1.7	6.3	tion with (010)	with a very definite	
C-1	3.3	4.3	2.8	1.1	0.56	0.40	1.2	3.9	slightly pre-dominating.	predominating of the (010).	
K-4	2.8	3.3	3.8	6.0	1.4	1.3	0.74	0.55	(110) and (010)	Increasing No. of	+ 8.3
K-8	1.7	2.2	3.8	4.8	1.0	0.74	0.45	0.46	planes normal to rolling direction with (110) predominating.	(110) and (010) planes normal to rolling direction. (110) still predominating.	- 1.7
M-4	3.1	4.4	1.7	1.7	0.39	0.70	1.8	2.6	(010) planes	Essentially	+ 98.0
M-8	3.6	4.4	1.4	1.5	0.36	0.33	2.6	2.9	normal to roll-	unchanged but	+ 69.0
N-12	5.0	5.4	1.3	1.3	0.29	--	3.8	4.2	ing direction	evidence of a slight	+139.8
N-14	3.8	3.8	1.3	1.4	0.44	1.22	2.9	2.7	with only a	increase of (010)	+120.9
N-21	3.8	4.6	1.3	1.6	0.40	1.30	2.9	2.9	slight amount of (110).	planes normal to rolling direction.	+145.8

$$* \bar{\rho}_{(010)} = \frac{\rho_{(020)} + \rho_{(040)} + \rho_{(041)}}{3}$$

Capsule Irradiations at the Materials Testing Reactor - R. S. Kemper

Uranium tensile specimens, enclosed with uranium split sleeves and canned with NaK as the heat-transfer medium in Zircaloy-2 capsules, were exposed in the MTR in a test to determine the properties of the material irradiated at beta or gamma phase temperatures.⁽²⁾ Three capsules were discharged January 16 after one operating cycle. The samples were exposed 14.85 effective days in the L-41 position. A flux monitor run made during exposure indicated that the unperturbed flux was $2 \text{ to } 3 \times 10^{14}$ nv. The samples are awaiting shipment to the Radiometallurgy Laboratory.

The Effects of Low Temperature Irradiation on the Properties of Uranium - R. S. Kemper and R. E. Hueschen

There is nothing new to report this quarter.

Composite Rods - R. G. Wheeler

The feasibility of making composite fuel element material by winding sheet stock into a tight spiral and swaging the resulting cylindrical element down to the desired diameter is being investigated. Initial exploratory work was done with a single metal instead of a composite of two or more. Copper sheet 0.010 inches thick was spiral wound, annealed and given a 65 per cent cold reduction inside a copper sheath. Density after swaging was 98 per cent of copper density. The rod sections were sintered in vacuum at 700 C (1290 F) and 880 C (1615 F) and in hydrogen at 910 C (1670 F). The per cent of the surface area between sheets bonded by grains linking one sheet with the next was 0.85, 49, and 0.7, respectively, for the sintering treatments listed above.

Porosity in the 880 C vacuum sintered specimens was confined to the space between the sheets whereas the specimens sintered at 700 C in vacuum and at 910 C in hydrogen were porous in the grain boundaries as well as between the sheets. Compacts of natural uranium were made by cold swaging a spiral wound 0.007-inch sheet to an 83 per cent reduction of area. The density of the resulting rod before sintering was 99.7 per cent of a similarly swaged solid rod of uranium. The effect of vacuum sintering these compacts is shown in Figures 3.1 and 3.2 which show the dilation of the rods in a direction parallel to the rod axis as a function of furnace temperature. Except for some shrinkage during sintering, the material behaves about the same in thermal expansion as does the swaged solid rod. After beta heat treating, the average grain size was about equal to the sheet thickness; however, none of the grains penetrated the bond between sheets

(2) All footnotes are referenced at the end of this section.

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FIGURE 3.1 DILATION OF SWAGED URANIUM SPIRAL COMPACT

FIGURE 3.2 DILATION OF SWAGED URANIUM SPIRAL COMPACT AFTER BETA HEAT TREATMENT

for either the high alpha or beta heat treatment. A qualitative test of the bond strength between the sheets revealed the bond to be strong in the uranium and weak in the copper even though the copper showed about 50 per cent of the bond between sheets was bridged by grains and uranium showed no bridging.

Flow Stress Recovery of Cold-Worked Dingt Uranium - R. S. Kemper

The flow stress recovery of dingt uranium sheet, cold-rolled to approximately 50 per cent reduction, is being studied for samples prepared longitudinal and transverse to the rolling direction. The data that have been obtained thus far are given in Table 3.3. Samples were prepared to obtain x-ray data for the as-rolled and base-annealed conditions for interpretation of the pronounced anisotropy in the properties of the material.

TABLE 3.3

TENSILE PROPERTIES OF DINGOT URANIUM SHEET

<u>Sample Condition</u>	<u>Relation to Rolling Direction</u>	<u>Flow Stress* 1000 psi</u>	<u>Elongation % in 1-Inch</u>	<u>Reduction of Area, %</u>	<u>Modulus of Elasticity 10⁶ psi</u>
50% C. W. as rolled	transverse	133.5	12	13	28
	"	132.5	12	15	29
	longitudinal	183.0	9	17	28
	"	187.5	6	17	29
annealed 600 C - 10 hr. vacuum >2 x 10 ⁻⁵ mm Hg	transverse	51.0	36	33	24
	"	52.7	37	30	32
	longitudinal	118.5	17	28	23
	"	118.5	16	27	26
annealed 200 C - 30 hr. vacuum >2 x 10 ⁻⁵ mm Hg	transverse	130.0	19	20	28
	longitudinal	175.8	10	20	29

* Flow stress at natural strain of 0.02 inch per inch.

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One group of samples was annealed in evacuated quartz capsules to determine whether this was a satisfactory means of carrying out the annealing studies. Pronounced differences in behavior were observed as indicated in Table 3.4. The longitudinal samples annealed in the quartz capsules also exhibited a change in type of fracture to a 45 degree failure.

TABLE 3.4
DUCTILITY OF ANNEALED DINGOT URANIUM

<u>Sample Condition</u>	<u>Relation to Rolling Direction</u>	<u>Elongation</u>		<u>Reduction of Area, %</u>
		<u>% in 1-Inch</u>	<u>% in 2-Inch</u>	
annealed 600 C - 10 hr. vacuum > 2 x 10 ⁻⁵ mm Hg	transverse	36	34	33
	"	37	33	30
	longitudinal	17	13	28
	"	16	14	27
annealed 600 C - 10 hr. evacuated quartz capsule	transverse	--	26	24
	"	--	23	32
	longitudinal	--	2.0	7
	"	--	2.5	4

Analyses for hydrogen, oxygen, and nitrogen are being made to determine whether gas impurities are responsible for the embrittlement of the material annealed in the quartz capsules.

The Effect of Irradiation on the Properties of Thorium - L. J. Chockie

The fabrication of specimens has been started for the program. There are no test results this quarter.

Reactor Structural Materials

Irradiation of Zircaloy-2 Tensile Specimens - R. S. Kemper

A total of twenty-four Zircaloy-2 tensile specimens were assembled in irradiation containers with graphite flux monitors for exposure in the MTR. (3) One group of twelve samples were exposed for two operating cycles in the L-47 position for an estimated fast neutron (> 1 Mev) exposure of approximately

(3) All footnotes are referenced at the end of this section.

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1.5×10^{20} nvt. These samples are awaiting shipment to the Radiometallurgy Laboratory. The second group of twelve samples was charged in the L-47 position March 19, 1956, and is scheduled for discharge April 30 with approximately the same exposure.

In-Pile Reaction of Zirconium with Selected Gases - R. G. Wheeler

There are no new data to report.

The Effect of Present Pile Atmosphere on Uncooled Process Tube Section - R. G. Wheeler

There are no new data to report.

Effect of Pile Atmosphere on Internally Cooled Process Tubes - R. G. Wheeler

The results of examination of a zirconium process tube removed from 100-H Pile are reported by W. S. Kelly under Radiometallurgy.

Zirconium Gas Reactions - L. F. Kendall

Previous investigations have indicated that vacuum annealing of oxidized Zircaloy-2 greatly accelerates the reaction rate during subsequent oxidation.⁽⁴⁾ This phenomenon has been confirmed in recent tests with zirconium which also show, as before, that the amount by which the reaction rate is increased is proportional to the length of the annealing time in vacuo. The two sets of data are plotted in Figures 3.3 and 3.4, respectively. A new aspect of this phenomenon appears in the latter figure: the uppermost curve indicates that although vacuum annealing before oxidation apparently does not affect the oxidation rate during the first few hours' exposure to air, the acceleration in the reaction rate after a second vacuum heat-treatment is aggravated. Extension of this effect to practical applications is premature since other variables may have influenced these data--e.g., grain size was undoubtedly larger in the specimens heated for the additional four hours while the hydrogen content may have been reduced. These effects will be investigated further.

Oxide Film Orientation - D. L. Gray and R. G. Wheeler

The relationship between the orientation of zirconium grains and the orientations of the oxide which forms on these grain surfaces (epitaxy) as well as the allotropic modification of the oxide determines the degree of misfit between the zirconium atoms in the metal and those in the oxide. In studying the oxidation of zirconium, both in the presence and absence of pile irradiation, these factors should be considered. Electron microscopic and electron diffraction examination of thin oxide films chemically stripped

(4) All footnotes are referenced at the end of this section.

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FIGURE 3.3 Effect of vacuum heating on reaction rate of Zircaloy-2 in air at 600 C (1110 F). All specimens were heated in air for six hours, then vacuum heat-treated at 600 C for the times indicated on each curve, and finally heated again in air.

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FIGURE 3.4 Effect of vacuum heating on reaction rate of sponge zirconium in air at 700 C (1290 F). All specimens were heated in air for four hours, then vacuum annealed at 700 C for the times indicated on each curve, and finally heated again in air. Specimens represented by top curve were vacuum annealed for four hours at 700 C before initial exposure in air.

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from zirconium, Zircaloy-2, and Zircaloy-3 show these films to be single crystals over any particular grain and the orientation of the oxide crystal differs from one grain to the next. The exact orientation of the oxide as affected by orientation of the zirconium, temperature of oxidation, method of stripping, and neutron flux was not determined in the initial work although the (111) plane of the oxide does appear to be closely aligned with the basal plane of the zirconium.

Recrystallization and Recovery of Zirconium Alloys - S. H. Bush, L. F. Kendall, L. J. Chockie and R. S. Kemper

The kinetics of recrystallization and recovery in zirconium, Zircaloy-2, and Zircaloy-3 are being determined to establish the optimum conditions of heat treatment during fabrication operations. Per cent cold work, temperature, time, and heat treat atmosphere have been selected as variables.

Initial studies were made to determine optimum conditions for process annealing. Sheet samples of Zircaloy-2 were prepared with an identical rolling and intermediate annealing sequence, one group with 650 C, one hour, vacuum anneals; and the other with 750 C, one minute, salt bath anneals. The final anneal in both cases was a 600 C, one hour, vacuum anneal since this anneal produces a dead-soft Zr-2 and Zr-3 with no perceptible grain growth. Samples of this material were corrosion tested in 680 F (360 C) demineralized water to determine whether salt bath annealing adversely affects the corrosion resistance. These tests have only run to 43 hours and in this time samples of both materials exhibit a tight, gray-black oxide and weight gains of approximately 15 mg/dm². Chemical analyses were made of Zircaloy-2 after vacuum and salt bath anneals, but before the final 600 C vacuum process anneal. The hydrogen content was essentially the same (17-20 ppm) after either anneal, whereas nitrogen was 12 ppm after vacuum and 33 ppm after salt bath annealing. The latter value is not considered excessive.

The fabrication of test samples of these materials was begun based on the results given above. Specimen material was produced by rolling 1/4-inch thick Zircaloy-2 plate and 0.205-inch thick Zircaloy-3 plate in controlled steps with intermediate salt bath anneals of one minute at 750 C. The anneals were followed by sand-blasting and acid pickling to remove any contaminated surface material. A standard base condition was established before introducing the cold work level by a 20 per cent reduction followed by vacuum process annealing one hour at 600 C. The final reduction produced three levels of cold work; 10, 25, and 50 per cent, with a sheet thickness of 0.062 inches. Approximately 100 samples, 6 inches long, and 0.350 inches wide, were prepared for each cold work level for both materials.

Creep Strengths of Cold Worked Zircaloy-2 - L. J. Chockie

There is nothing new to report this quarter.

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Fundamental Studies

Metallography of Uranium - T. K. Bierlein, B. Mastel, and D. L. Gray

The intrinsic value of electron microscopy as compared to optical microscopy lies in its increased resolution and depth of focus. In the analysis of the effects of reactor irradiation on the microstructure of uranium, the high resolution of electron microscopy may reveal fission product concentrations at grain boundaries, micro-voids, as well as other microscopic structural changes which can not be observed by optical methods. Electron microscopy is, therefore, a potentially valuable tool.

Since many of the irradiation effects on uranium originate on an atomic scale and can manifest themselves on a micro and macro scale, metallographic studies should cover the entire magnification range. Recent experiments on non-irradiated, beta heat treated uranium indicate that Faxfilm replication provides a simple means for conducting optical and electron microscopy on the same area.

The specimen to be examined is first marked on its polished face to provide a reference for orientation and distance. The specimen is then cathodically vacuum etched and replicated twice with Faxfilm. One of the Faxfilm replicas is prepared for optical microscopy and the other for electron microscopy, as indicated in HW-34390.⁽⁵⁾ A small area, 1 mm x 1 mm, is cut from the latter replica such that its orientation and distance from the reference mark is known. After solution of the Faxfilm in acetone, the thin film is picked up on a supporting grid and examined in the electron microscope. The film within any grid opening is photographed and this area is then found on the second, duplicate Faxfilm replica by optically scanning a square millimeter area. Once the proper area has been found, any grid opening containing a suitable replica film can be photographed on the electron microscope and correlated with optical micrographs of identical areas.

Incorporation of this technique in the metallographic study of highly radioactive specimens introduces no complications since remotized cathodic vacuum etching and Faxfilm replication has been used satisfactorily on irradiated uranium. Only the asymmetric reference mark is required and can be produced by a remotized hardness indenter or a micropunch. Application of this technique to irradiated materials should increase the value of metallographic investigations.

Figure 3.5 contains micrographs of non-irradiated uranium subjected to this examination technique. Several characteristics in the metallography of uranium warrant emphasis. The macro structure of uranium is a function

(5) All footnotes are referenced at the end of this section.

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-3. 15-

HW-42390

Uranium - 3X

Uranium - 750X

Replica - 750X

Electron Micrograph - 3,000X

Electron Micrograph - 30,000X

FIGURE 3. 5

Correlation of Optical and Electron Micrography - Cathodically Vacuum Etched, Beta Heat Treated Uranium.

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of the illumination used, and will change as the angle of illumination is varied. A macro grain will change in size and shape similar in many respects to the change in the grain structure which occurs during rotation of a uranium specimen illuminated with polarized light. Contrast in the microstructure under bright field illumination can easily be enhanced by heat tinting which produces a multicolored microstructure. Micrography with polarized light requires a multiplicity of photographs of a given area to adequately reveal all the microstructure, whereas oblique bright field illumination of a cathodically vacuum etched specimen requires at most two photographs corresponding to two angular positions of the oblique beam of light. In Figure 3.5 the bright field micrograph of uranium was obtained after the specimen surface had oxidized to increase grain contrast and is compared with the micrograph of the replica prepared immediately after the specimen had been cathodically vacuum etched. As can be seen, the replica is a faithful reproduction of the uranium surface. The severity of the pits in the uranium is exaggerated by the oblique illumination. The extent to which the resolution is limited in optical microscopy is illustrated by a comparison with the identical area photographed in the electron microscope. Combined use of optical and electron micrography is beneficial and should contribute to the proper understanding of radiation induced changes in uranium.

Electron Microscopy of Irradiated Uranium - T. K. Bierlein and B. Mastel

Remotized cathodic vacuum etching and Faxfilm replication have been applied to a specimen of uranium irradiated to 1.5×10^{20} nvt, and electron microscope studies have been conducted. As the micrographs in Figure 3.6 indicate, a variety of grain structures exists on the specimen surface. Perhaps this situation is to be expected, since a large number of irradiation variables may independently and collectively cause major changes in the microstructure of irradiated uranium. The situation is further complicated by the fact that the sensitivity of cathodic vacuum etching as applied to irradiated uranium may be sufficient to reveal even slip and cross-slip caused by localized stress fields.

The upper left hand micrograph of Figure 3.6 certainly provides evidence of extensive twinning; angular variations in the twin bands may be due to perturbations introduced by kinking or deformation bands. The upper right hand micrograph contains a grain the appearance of which is indicative of slip and cross-slip, a possibility which will be investigated. Some of the structure in the lower micrographs may arise from a domain structure and polygonization as a result of kinking. The boundaries of these domains would serve as sinks for fission products, vacancies, and the like, although no particular structure at these boundaries is discernible.

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FIGURE 3. 6

Electron Micrographs Illustrating the Microstructure of Uranium Irradiated to 1.5×10^{20} nvt. Cathodic Vacuum Etch; Remote Fax-film Replication. 3,500X

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The need for a reference or standard for evaluating microstructural changes in irradiated uranium is great. An approach to this problem has been made, in that metallographic specimens, which have been studied and photographed prior to canning in NaK and irradiation, will soon be examined, and changes in the microstructure of particular areas will be established by both optical and electron microscopy methods.

Deformation and Fracture in Uranium - T. K. Bierlein and R. E. Hueschen

The fundamental cause and mechanism of the deformation and fracture in irradiated fuel elements is not known, and the reasons for decrease in ductility and ultimate strength and increase in yield strength have not been established. Since tensile, bending, and impact properties are directly related to microstructure an in situ study should permit direct correlation. Once this correlation is established, physical significance can be ascribed to microstructural appearance and property values.

Since it is necessary to understand the microscopic behavior of unirradiated uranium under stress before the effects of irradiation can be studied, prototype equipment is being designed and developed. A hot stage microscope containing a microtensile fixture has been designed and is now ready for construction.

Further study of the specimen surface shown in Figure 3.7 has revealed a unique orientation of this particular grain. This specimen was prepared by mechanical polishing, electropolishing, and cathodic vacuum etching as previously reported.⁽⁶⁾ As noted, some etch pits are parallel to twin bands, and others are parallel to some other crystallographic plane. One surface pole loci analysis of the traces contained in this grain indicated that this surface of the grain is parallel to the (001). The predominant twins are {130}, the etch pits delineate {172} or {176} twins, and the straight boundary within this grain is a (100) kink boundary. The present explanation for the appearance of these etch pits is as follows. Normally cathodic vacuum etching removes surface atoms from the metal at a rate proportional to the energy of the individual atoms. If, however, oxygen or similar reactive contaminants are present in the inert gas which is used for etching, simultaneous chemical attack occurs. The rate of the combined attack is preferential on particular planes and in certain directions. Dislocations located at the slip, twinning or kinking planes etch preferentially and account for the observed pits in this particular grain of an annealed uranium specimen.

(6) All footnotes are referenced at the end of this section.

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FIGURE 3. 7

Optical Micrograph of a Cathodically Vacuum Etched Specimen of Annealed Uranium from Which an Analysis of Crystallographic Planes Has Been Made. 1,500X

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Diffusion Studies - D. R. Green

Laboratory investigations of the diffusion of uranium into zirconium and AlSi are continuing to provide a basis for interpreting in-pile experiments currently in progress.

Previous studies have shown that the diffusion zone in mechanically constrained U/Zr couples is very non-uniform, apparently due to non-uniform surface contact. Vacuum hot pressing equipment capable of producing a slight yielding of the specimens and subsequent uniform interface contact during the diffusion anneal is now being used in the U/Zr studies. Two U/Zr couples are being vacuum annealed at a temperature of 450 C (842 F) and a pressure of 17,500 psi. Pre-bonding a couple by means of a thin, uniform initial diffusion zone should overcome the problem of poor interface contact during the diffusion anneal. A U/Zr couple was pre-bonded by hot pressing at 700 C (1292 F) and 2100 psi, and is currently being vacuum annealed at 450 C (842 F) to determine the feasibility of using this type of couple.

Two mechanically constrained U/AlSi couples were vacuum annealed 673 hours (28 days) at 255 C (491 F). Preliminary observations indicated that the uranium had diffused approximately 0.003-inch into the AlSi from the original U/AlSi interface.

Several methods for determining the maximum extent of diffusion of uranium into zirconium and AlSi are being compared. The method currently being investigated utilizes systematic observation of the extent of diffusion at a number of points throughout the interface. Statistical methods are then applied to determine the maximum extent of diffusion. No further results of the in-pile studies are available at this time.

Fission Product Mobility Experiments - J. Martin Tobin

The diffusion coefficient of krypton gas in silver at 735 C has been measured this quarter. Its value is $D_{735} = 4.3 \times 10^{-8} \text{ cm}^2/\text{sec}$. LeClaire and Rowe⁽⁷⁾ obtained the value of $D_{735} = 5.7 \times 10^{-9} \text{ cm}^2/\text{sec}$ for the diffusion of argon in silver at the same temperature. The value of the krypton diffusion coefficient was obtained from the following data:

<u>Ampoule Volume</u>	<u>Corrected Gas Activity*</u>	<u>γ</u>
$V_6 = 4.13 \text{ cc}$	9,270 counts/minute	1.00
$V_5 = 3.87$	8,970	0.87
$V_4 = 3.86$	8,560	0.81
$V_3 = 3.72$	7,000	0.69
$V_2 = 3.16$	7,080	0.63
$V_1 = 3.84$	0	0
$V_0 = 28.20$	-	-

* (at 0.150 Mev, corrected to standard time).

(7) All footnotes are referenced at the end of this section.

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The volume contained in each ampoule was a known fraction of the gas in the system. This amount was corrected for the amount of gas removed in previous samples. The fraction of gas diffused out of the silver in times t (sec) is defined as γ , and the silver thickness as h . The diffusion coefficient is obtained from the following equation:

$$\gamma = 1 + \frac{4}{\pi} \sum_{s=1}^{\infty} \frac{(-1)^{s+1}}{2s-1} e^{-(2s-1)^2 \frac{\pi^2 Dt}{h^2}}$$

γ	t
1.00	fused silver
0.87	3850 sec.
0.81	3050
0.69	2250
0.63	2000
0	0

Several sets of krypton gas samples have been collected and are awaiting irradiation in the pile. The irradiations have been delayed several weeks while the slug design has been changed. This was necessary in order to prevent contamination of the pile basin area.

The method of radioanalysis which was developed for analysis of krypton gas samples was used successfully this quarter for the analysis of xenon gas. This will enable us to perform experiments in diffusion of xenon gas in metals, as well as krypton. In fact, the use of xenon gas has several advantages over the use of krypton. Xenon is six to eight times more abundant as a fission product. Also, the analysis of xenon is much easier due to the higher neutron capture cross-section (about 35 barns total cross-section) and longer half-life (about 18 hours for xenon 125). The transfer of radioxenon from quartz to glass ampoules can be delayed for 18 hours till the quartz radioactivity has decayed and is quite safe to handle without radiation monitoring service.

Analysis of the gamma radiation from radioxenon 24 hours after leaving the pile shows there are many gamma emitters present. The 0.19 Mev gamma radiation peak was chosen for the analysis. The activity of this peak for several samples of gas containing known amounts of xenon shows that the activity is directly proportional to the concentration.

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<u>Ampoule</u>	<u>Xenon Content</u>	<u>Activity* (0.19 Mev)</u>
J1	3.62×10^{-10} gram atoms	1.4×10^2 counts/min.
J2	1.56×10^{-9}	6.1×10^2
J3	1.19×10^{-8}	4.62×10^3
J4	3.27×10^{-8}	1.24×10^4
J5	4.20×10^{-8}	1.63×10^4
J6	None (He alone)	Not detectable

*All activities corrected to $t = 0$.

Calculations are being made in order to determine the capture cross-section of the xenon 124 isotope (0.096 per cent abundant in natural xenon). The data collected incidental to the above irradiation of xenon gas are sufficient to calculate this physical constant.

Effects of Irradiation on the Crystal Structure of Zirconium and Molybdenum - W. V. Cummings and E. C. Watts

Zirconium specimens irradiated in the MTR to exposures of 6.5×10^{18} and 6.5×10^{19} nvt fast flux (> 1 Mev) have been given preliminary examination by x-ray diffraction. Diffraction patterns obtained with the precision back-reflection focusing camera show no change in lattice spacings for the low exposure sample and only a slight shift to larger values for the higher exposure sample. The calculations are incomplete at the present time. The microstructure of the two samples compared to the unirradiated material indicates there are no significant changes. Other samples with higher and intermediate exposures are being prepared for study.

X-ray Diffraction Studies of Radiation Damage to Uranium - W. V. Cummings

The specimens for this experiment were originally scheduled for irradiation in a Poison Column Control Facility. (8) Scheduling and charging difficulties have persisted over a period of two years. Arrangements are now being made to irradiate the test pieces in a Snout facility in hopes that a charging or discharging during operation can be more easily accomplished.

Neutron Irradiation of Sapphire - R. G. Wheeler

An unclassified document entitled, "Neutron Irradiation of Sapphire (Al_2O_3)" HW-42022 has been released.

(8) All footnotes are referenced at the end of this section.

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Evacuatable Hot Stage Unit - T. K. Bierlein, R. E. Hueschen and D. C. Kaulitz

A proper understanding of the mechanism of deformation and fracture in uranium as a result of reactor irradiation requires a knowledge of the mode of deformation and fracture in unirradiated uranium as a function of temperature and stress. This type of study can be conducted most efficiently on a microscopic scale in which the deformation and propagation of cracks can be observed directly in non-irradiated uranium of various histories. Since uranium is chemically reactive, elevated temperature tests must be conducted in an inert atmosphere or in a vacuum. The latter has been selected for use; this requires proper design of an enclosure which will permit microscopic examination of the specimen during the period stress is applied.

Figure 3.8 depicts the basic components of the evacuatable hot stage unit which has recently been designed for construction. Tensile stresses are to be applied to the specimen by means of an hydraulic cylinder such that the force on the specimen can be determined to some degree of accuracy at any point in the deformation test. Specimen geometry is such that the maximum stress is applied over a small region, about 0.0025 in.², which can be scanned with an 8 mm microscope objective. Heating of the specimen will be achieved by passing a low voltage high current through the specimen such that the "gage" length of the specimen will be the hottest section. Strain in the gage section can be ascertained by relative displacements of ruled lines on the specimen surface.

Preselecting X-ray Goniometer - T. K. Bierlein, D. C. Kaulitz and R. E. Hueschen

The behavior of polycrystalline uranium as a function of applied stress is dependent on the orientation of individual grains which during plastic straining interact and cause gross changes in the overall shape of a specimen.

Since slip, twinning, and kinking, the normal deformation modes in uranium, can be identified within a grain if its orientation is known, an x-ray back reflection goniometer with provisions for metallographic preselection of a particular grain has been designed. The basic unit is similar to that described by Cahn in AERE-M/R-774,⁽⁹⁾ but incorporates two goniometer arc motions perpendicular to the incident x-ray beam. These motions, it is hoped, will facilitate Laue orientation studies and permit identification studies of isolated phases by Debye methods. Figure 3.9 depicts the general design of this x-ray goniometer, the intended major use of which will be to enhance the study of deformation and fracture in uranium.

(9) All footnotes are referenced at the end of this section.

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FIGURE 3.8 EVACUABLE HOT STAGE UNIT WITH PROVISIONS FOR CONTROLLED DEFORMATION OF TEST SPECIMENS

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FIGURE 3.9 GONIOMETER FOR METALLOGRAPHIC AND X-RAY APPLICATIONS

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- (8) Baars, R. E., Cummings, W. V., P.T. No. 105-519-SI, Irradiation Service Request No. HAPO 108, Studies of Uranium Crystal Structure Damage Using Low Irradiation Temperatures, HW-31163, March 22, 1954, (SECRET).
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PILE METALLURGY - J. J. CadwellFuel Element FabricationSwaged Oxide Fuel Elements - F. B. Quinlan, W. E. Roake

Irradiations carried out at other sites have shown that the release of gaseous fission products by irradiated UO_2 is a function of the surface area of the oxide. Material of high density exhibits greatly enhanced retention. The goal of the present work is production of densities of at least 90% of theoretical by an economical method. Material of this density is presently produced by high temperature sintering. Attempts are being carried out to produce high density UO_2 by jacketing UO_2 powder in stainless steel and reducing the diameter of the piece by swaging. In the latest test, the test piece was cold swaged to a reduction of 55 percent. One section of this piece was then hot swaged at 750 C to a further 44 percent reduction, and the other section cold swaged to 42 percent reduction. Sectioning the hot swaged sample showed that the original UO_2 powder had formed coarse crystals of metallic appearance. This material was very hard and friable, and contained many large cracks. The apparent density of the sample, including shrinkage cracks, was 86% of theoretical. If another reduction-pass had been made on the sample the shrinkage cracks would probably have been eliminated and the apparent density considerably increased. The hot swaging method appears to have great promise as an economical approach to the production of high density UO_2 . The cold swaged sample appeared to have partially transformed. It contained powder and numerous small needle crystallites. The apparent density of the sample was 78% of theoretical.

During the past month, three capsules were hot swaged to obtain preliminary indications of the effect of temperature on the degree of oxide compaction. Run of the plant Mallinckrodt UO_2 was tamp packed (to about 45 percent theoretical density) into sections of schedule 40 304 stainless steel pipe fitted with threaded end plugs which were subsequently welded in. Three such sections were swaged at about 600 C, taking a diametral reduction of approximately 0.050" per pass. Cross sectional area (oxide) reductions of 39.8, 47.1, and 54.1 percent resulted in oxide densities of 66.9, 73.9, and 83.6 percent of theoretical. The experiments were stopped at this point due to difficulties encountered in swaging over the welds, and the capsules were sectioned for examination. The oxide compacted to 83.6 percent of theoretical density contained many small crystals of UO_2 produced during swaging. Surface area of the oxide was measured by nitrogen adsorption and was found to be essentially unchanged by the swaging.

Attempts are being made to produce a thin walled uranium oxide fuel element. The wall or jacketing materials considered are aluminum,

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zirconium, and 18-8 stainless steel. Tests are directed toward making an oxide element in one series of swaging operations. These swaging operations should produce, from an assembly of a metal cylinder, uranium oxide (powdered UO_2) and two end plugs, a finished totally enclosed high density fuel element.

Swaging cannot be done directly on the thin walled jacket material. The high compressive stresses required for compaction of the oxide could not be retained by such a weak jacket. This jacket is therefore reinforced by an outer thick walled cylinder of mild steel. The mild steel cylinder is plugged at each end to retain the fuel element components. This entire assembly is then swaged to the proper size, after which the steel is removed mechanically or chemically.

First attempts, using thin walled aluminum tubing and with Al_2O_3 instead of UO_2 , show considerable promise. Al_2O_3 is being used to simplify and cheapen the exploratory phases of this new technique. Zirconium tubing will be tried when it becomes available. Figure 4.1 shows a transverse section of the Al_2O_3 , aluminum wall, and steel jacket. Figure 4.2 shows a section of the aluminum tube and Al_2O_3 after removal of the steel jacket. This piece has undergone a 33% reduction in cross sectional area.

Cluster Fuel Element Testing - G. T. Geering

Cluster fuel elements meet many of the requirements of high temperature recirculating water reactors. Hanford fuel elements cause rapid and severe flow blockage when ruptured at 260 C in standard process tubes. Four rod clusters containing 54 percent of the uranium in a standard slug have been ruptured in ELMO-4 recirculating loop at high temperature. No appreciable flow reduction was measured in these experiments. In addition to these rupture safety considerations, a cluster fuel element has greater heat transfer surface than a standard slug, and can operate at higher specific powers for a given uranium temperature.

Several experimental fuel elements of cluster geometry have been built during this quarter. The goal of this work is to design, test and build fuel elements for the operation of the KER recirculating facility which is scheduled to operate beginning mid-July, 1956. Part of the testing has included building a few fuel elements for irradiation in H-pile loop. Two of these four rod clusters containing uranium will be irradiated during the next quarter.

Tests were run in 305 pile to determine the flux distribution in the proposed KER fuel element. The results are reported in HW-42231 by

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FIGURE 4.1 Section of fuel element in steel swaging jacket

FIGURE 4.2 Section of fuel element removed from jacket

Photographs Unclassified

AEC-GE RICHLAND, WASH.

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A. W. Theile. Using this flux data, the power generation of a KER fuel element with stainless steel cladding is about 1.02 times greater than that generated in a solid slug in an adjacent tube. This calculation assumes that the KER tube will be Zircaloy-2. The design of a suitable support spider to locate a cluster fuel element in a horizontal process tube is a major problem. The spider must be rigid enough to maintain alignment, flexible enough to prevent large discharge forces in the case of fuel distortion, and must come apart under the weight of each fuel assembly as it is discharged. Figures 4.3 - 4.6 illustrate two possible designs.

Internally and Externally Notched, Cored, Unbonded Fuel Element Experiment (GEH-4-4) - T. W. Evans

The specimens remain at the MTR. They will be irradiated when space in the testing facilities is available.

Fuel Elements

Combined Fuel-Target Elements - J. W. Goffard

There is nothing new to report on the development of these elements.

Hanford Size Uranium-Magnesium Fuel Elements - J. W. Goffard

The HAPO size U-Mg fuel element that is to be operated through one reactor cycle at the MTR before the production test charge will be loaded at Hanford, is still in storage at the MTR. It will not be possible to reload this piece in the GEH-4 facility until ventilation modifications are completed.

Uranium-Magnesium Fuel Material for Cluster Fuel Elements - J. W. Goffard

Uranium-magnesium fuel material is being prepared for cluster geometry fuel elements. The fuel material will be 8" long and about 1/2" diameter, and the clusters will consist of Zircaloy clad fuel spaced with stainless steel dovetailed spiders. One hundred and fifty pounds of uranium shot in the size range 2000-150 microns is available for fabrication of the fuel material. This shot was received from Mallinckrodt and is believed to be a by-product of uranium bomb reduction operations. Irradiation of the U-Mg fuel bearing clusters will be accomplished initially at the MTR followed by larger scale testing in a HAPO recirculating loop facility.

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FIGURE 4.3

KER Fuel Element - cross section of tube shown
Photograph Unclassified

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FIGURE 4.4

KER Fuel element for ribbed tube

Photograph Unclassified

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FIGURE 4.5 Fuel element for H-loop test
process tube shown

FIGURE 4.6 Fuel element for H-loop test
spider partially unlocked -
two of eight rods shown

Photographs Unclassified

AEC-GE RICHLAND, WASH.

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Fuel Element EvaluationThermal Stress Calculations for Solid Uranium Fuel Elements Operating Between 100-350C and 350-600 C - K. R. Merckx

Realistic calculations for the thermal stresses and strains in fuel elements must use a model of mechanical behavior for uranium which includes the time and temperature effects of plastic deformation. An analysis of the thermal stresses and strains, which uses a time and temperature dependent model, ⁽¹⁾ has been completed for solid fuel elements operating between 100 C to 350 C and 350 C to 600 C. For both cases, the heat generation is assumed to increase uniformly for 25 minutes - maximum temperature increases 10 C/min - then the heat generation is held constant for 700 minutes. Calculations for the above temperature ranges point out the differences in thermal stress build-up and relaxation for high and low temperature operation.

The calculated stresses after power increase and 700 minutes' relaxation are given in the following table:

COMPARISON OF THE STRESS QUANTITY, σ_o ,
WITH DIFFERENT AMOUNTS OF STRESS RELAXATION

		<u>Case 1</u>	<u>Case 2</u>
Surface Temperature		100 C	350 C
Central Temperature		350 C	600 C
From Elastic Equations (independent of time, t)	σ_o surface (psi)	90,200	30,200
	σ_o center "	45,100	15,100
Immediately After Power Increase (t = 25 min)	σ_o surface "	36,400	15,600
	σ_o center "	18,400	920
After a Period of Stress Relaxation (t = 722 min)	σ_o surface "	31,200	11,700
	σ_o surface "	14,300	0

The stress quantity σ_o is the von Mises yield criterion for materials which are triaxially loaded. For a uniaxial tensile test, σ_o equals the tensile stress in magnitude. The von Mises yield criterion is the stress quantity which is used to compare the effects of plastic yielding and ultimate strength of ductile materials triaxially loaded to the test results obtained from tensile tests. The radial distribution of σ_o for case 1 and case 2 at t = 25 min and t = 700 min is given in Figure 4.7. The build-up and relaxation of σ_o at the center and the surface for case 1 and case 2 is given in Figure 4.8.

(1) All footnotes are referenced at the end of this section.

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For the low temperature case, case 1, the stress relaxation occurring during power increase accounted for most of the stress relaxation. The stresses calculated with elastic equations are reduced by a factor of 2.5 at the surface and 3.15 at the center when plastic flow of the uranium is considered during the period of power increase (Figure 4.8). Further reductions of 14% in the surface stress and 22% in the central stress occur during the 700 minutes' operation at constant power generation (Figure 4.7).

For case 2, the high temperature case, the central thermal stresses are annealed out within five minutes after steady state temperature is reached. In fact, relaxation effects at higher temperatures cause the central stresses to decrease during the period of increasing power generation (Figure 4.8). The central stress reaches its highest value, $\sigma_0 = 6,320$ psi, at $t = 11.4$ min and $T_{\text{center}} = 464$ C.

Figures 4.7 and 4.8 show that stress relaxation must be considered at operation of the uranium fuel elements, especially above 400 C. Thus, if the whole fuel element can be kept at high (above 400 C) temperatures continually, these calculations predict that the thermal stresses in a fuel element would be completely annealed out.

In-Pile Testing of Unbonded Fuel Elements - D. L. Zimmerman,
J. E. Minor

Unbonded slugs have been irradiated to investigate their resistance to cleavage failure and the extent to which aluminum-uranium interaction occurs in-pile. The uranium slugs were canned in aluminum by the point closure technique. Oxidation of the uranium surfaces was the only diffusion barrier present.

Fabrication of these fuel elements, irradiations, and post-irradiation examination have been discussed in previous reports of this series. (1, 2, 3, 4, 5, 6). No further examination time has been available during this quarter. During the coming quarter an enriched cored slug which operated at 70 kw/ft will be sectioned and etched to determine what portion of the slug operated in the beta phase. Examination of a natural uranium cored slug which operated at 55 kw/ft has shown that this slug did not attain beta phase temperatures. Information from the enriched slug will permit an estimate to be made of the temperature drop which occurred across the unbonded aluminum-uranium interface.

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FIGURE 4.7

EQUIVALENT TENSILE STRESS DISTRIBUTION
FOR A SOLID FUEL ELEMENT
(For Two Temperature Ranges And Different Periods Of Stress Relaxation)

FIGURE 4. 8

EQUIVALENT TENSILE STRESS BUILD-UP AND
DECAY FOR A SOLID FUEL ELEMENT
(Two Temperature Ranges.)

The Examination of Cored and Solid AlSi Bonded Slugs Irradiated in the MTR (GEH-4-1) - C. L. Boyd and T. W. Evans

The data obtained from examination of these slugs during the current quarter is reported in the Radiometallurgy section of this report.

Notched Solid Slug Experiment (GEH-4-2) - T. W. Evans, D. P. O'Keefe

There is nothing new to report.

In-Pile Testing of Insulated Fuel Elements - J. E. Minor, D. L. Zimmerman, W. S. Kelly

Insulated fuel elements are being irradiated to study the performance characteristics of Hanford geometry fuel elements in which the uranium is maintained at elevated temperatures during irradiation. Fabrication, irradiation, and preliminary examination of test slugs have been discussed in previous reports of this series.

In the preceding report of this series, it was reported that the second loading of insulated slugs in the MTR operated at 107-110 kw/ft rather than the 85 kw/ft for which they were designed. Calculations based on this higher power level showed a maximum central temperature in the uranium of 1160-1190 C. The three slugs from this loading have now been decanned and examined. An end view of slug N-1 was shown in the previous report. Frozen droplets of previously molten uranium were visible at the base end of this cored slug. Figure 4.9 shows two longitudinal sections from this slug. There is little evidence of melting in the top one-third of the slug. Rather extensive melting occurred in the middle of the slug, and the bottom one-third of the original 3/8 inch axial core was completely plugged.

End views of slugs G-2 and G-3 are shown in Figure 4.10 and 4.11. Melting had occurred in G-3 to a greater extent than in N-1. G-2, however, was still an open cylinder of uranium and no evidence of melting was visible. The variation in melting behavior between these three slugs is probably related to variation in flux intensity along the column of slugs. The three slugs were loaded vertically in the Testing Facility. In the first day of operation this flux variation would cause the bottom slug of the loading to operate at higher power than the other two, and the top slug to operate at the lowest power.

HAPO Fuel Element Testing Facility in the MTR - D. C. Kaulitz, F. B. Quinlan, T. W. Evans

The GEH-4 facility at MTR has caused a contamination problem because of its inability to contain fission gases resulting from ruptured slugs.

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Upper Third of Slug

areas
of melting

Remainder of
core plugged

Middle Third of Slug

FIGURE 4.9 Longitudinal Section of N-1
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FIGURE 4.10 Base End of G-2 After Removal
of End Caps

1.5 X

FIGURE 4.11 Base End of G-3 After
Removal of Can

1.5 X

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After the effluent water is discharged into the hot waste disposal system at the MTR, there is no way of being assured that the fission gas will be contained within the system. The GEH-4 facility is the only one at the MTR that is not a closed, recirculating loop. In order to correct the gas contamination problem a resin bed and "hot" hold-up tank are being installed on the effluent line. When a rupture does occur the resin will remove the fission products and the gas will be drawn off by negative pressure from the holdup tank, to their hot exhaust system. The storage tank will be of sufficient volume to handle the water required to cool the slugs after the reactor has shut down due to a rupture in the facility.

Design and fabrication of the above improvements cannot be completed until June 1. In the meantime, a series of non-rupture type tests are being carried out. One of these tests is to determine the specific amount of gamma heating in specimens in the GEH-4 facility. The exchange of thermal energy within the GEH-4 facility at the MTR has been uncertain for some time. Two of the principal factors involved are the generation of heat within the slugs from the intrinsic gamma field in the MTR and the heat transfer to the loop cooling water from the higher temperature reactor water. To obtain a better understanding of these factors for more exact calculations of temperature distributions within each experimental charge, an aluminum clad lead slug containing thermocouples was installed in the GEH-4 facility. After the reactor reached full power, calculations showed that the gamma heating was 1 watt/gm or for a solid uranium slug (Hanford Standard) would be 5 KW/ft.

Reaction of Fuel Materials with High Temperature Water - J. E. Minor, D. C. Kaulitz, R. G. Post

A study is being carried out of the kinetics of the reaction between hot flowing water and uranium heated 400 - 500 C above water temperature. The principal object of the study is to evaluate the hazard arising from the reaction between uranium heated to 500 - 700 C and water at temperatures from 150 - 300 C.

Experiments involving reaction between bare uranium rods, heated or unheated, and hot flowing water have been described in previous reports of this series (5, 6). Initial experiments involving "canned" specimens have also been described. The experiments with canned specimens have been continued in an endeavor to observe the corrosion behavior of uranium sheathed in aluminum when the uranium is held at high temperature during the corrosion reaction.

The type of can currently being used is illustrated in Figure 4.12. The metal can is fabricated in three pieces, the end pieces being stainless steel and the central "spool" aluminum. The central spool is electrically

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insulated from the end pieces by mica gaskets. Each assembly screw (not illustrated in Figure 4.12) is insulated from the end pieces by an anodized aluminum washer and by porcelain insulators. A spool of Alsimag inserted in the aluminum spool insulates the aluminum thermally and electrically from the uranium specimen. After installation in the reaction chamber the can is defected by an 0.065 inch hole drilled through the aluminum and Alsimag spools. This assembly permits electrical heating of the uranium during exposure to water. The specimens tested have been unalloyed uranium, not bonded to the can. When an assembly of this type is exposed to 300 C water and the uranium is heated only to temperatures not exceeding 400 C, hydrogen generated at the point of initial water contact attacks the uranium in a rapid and extremely non-uniform manner. Figure 4.13A shows the appearance of the uranium of a canned specimen exposed to 300 C water for 12.5 minutes.

In a later experiment a canned specimen was exposed to 300 C water for about 19 minutes. The uranium of the specimen was held at 720 - 750 C during the reaction. Figure 4.13B shows the appearance of the uranium of this specimen after reaction and cleaning. Pitting of this specimen by hydride formation is very nearly absent. Further experiments are planned to determine the lowest uranium surface temperature necessary to minimize hydride formation during the corrosion reaction. Results of these experiments will indicate whether an insulated slug operating within practical temperature limits can be expected to resist hydride formation if the slug cladding should fail in service.

Insulated Slug Rupture Experiment (GEH-4-7) - T. W. Evans

The successful irradiation of two insulated fuel element specimens to approximately 1000 MWD/T (see previous reports of this series) has prompted further study of the behavior of specimens of this design under irradiation. Because the value and usefulness of a fuel element depend strongly upon the failure characteristics of the element, it is important that some effort be made to define these failure characteristics.

An experiment had been planned for the HAPO fuel element testing facility in the MTR in which three insulated fuel elements, each with a different "built-in" failure mechanism, would be irradiated until a failure occurs. In January the operation of the testing facility had to be suspended temporarily (see elsewhere in this report) and thus this experiment has had to be postponed until space is again available in the testing facility.

In-Pile Testing of the Uranium-Magnesium Matrix Fuel Material - M. D. Freshley and W. S. Kelly

The properties of a high burnup fuel material which consists of discrete uranium particles embedded in a magnesium or magnesium-silicon alloy

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FIGURE 4.12 Canned Alsimag Insulated Specimen for Corrosion Experiments

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- A. Run 29 Uranium temperature 350-400 C
Water temperature 300 C
Exposure time 12.5 minutes

- B. Run 35 Uranium temperature 720-750 C
Water temperature 300 C
Exposure time 19 minutes

FIGURE 4.13 Canned Specimens after corrosion and cleaning. Central aluminum spool removed by dissolving in caustic.

matrix are being investigated for suitability as a reactor fuel material. This fuel material, which has been described in previous issues of this series of reports, has properties that are desirable for successful reactor operation. The chief advantages are increased thermal conductivity, dimensional stability, and improved resistance to radiation damage.

Two uranium-magnesium capsules that were irradiated to about 20,000 MWD/T of uranium have now been decanned, examined, and bend tested. The external appearance of the two capsules, which had been irradiated to two per cent burnup of the total uranium atoms, was the same as the previous capsules in that they appeared unchanged following exposure to this level. No distortion of the samples was apparent after decanning.

It was difficult to decan the sample that contained a pure magnesium matrix. The can was removed with a parting lathe. One end of this sample was fractured, but the remaining length of sample was sufficient for bend testing. The other sample, which had a matrix of magnesium-1.4 w/o silicon, was easily removed from the can by tapping. Both samples were bend tested by center loading over a one-inch gage length. Maximum loading at failure for the sample which contained a pure magnesium matrix was 940 pounds, with a maximum deflection at the midpoint of the gage length of 0.009 inches. The maximum load of the magnesium-silicon matrix sample was 970 pounds with a maximum deflection of 0.0075 inches.

The load-deflection curves for the samples with two per cent burnup have about the same slope as the curves for the previous samples, but the ultimate load at failure has decreased slightly. Also, the load-deflection curves are straight lines which indicates that the material is not yielding plastically. This effect, however, apparently saturated at about 0.10 per cent burnup. Extended pile exposure and additional radiation damage to the uranium have not greatly affected the properties of the fuel material. The samples investigated were dimensionally stable and free from distortion after being irradiated to two per cent burnup of the total uranium atoms.

Fuel Materials

Powder Metallurgy U-Mg Fuel Material - J. W. Goffard

Five powder metallurgy U-Mg fuel material specimens prepared by KAPL have been encapsulated in aluminum and shipped to the MTR for irradiation. Figure 4.14 shows the five specimens prior to canning. Item 23-H of the figure is the control piece for items 25-H and 30-H. Control pieces for 26-H and for 44-H and 45-H are also available. Since the properties of the U-Mg fuel material change most rapidly during the early stages of burnup, the test specimens will be exposed to only 1000 and 3000 MWD/T

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FIGURE 4.14 KAPL Powder Metallurgy Fuel Material Specimens before Encapsulating in Aluminum for Irradiation in the MTR. Item 23-H is the control piece for 25-H and 30-H. Other controls are not shown.

(1.5X)

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prior to post-irradiation examination at the HAPO radiometallurgy facility. Table 4.1 summarizes the data on the powder metallurgy U-Mg specimens.

TABLE 4.1

DATA ON KAPL POWDER METALLURGY U-Mg FUEL MATERIAL SPECIMEN

<u>Speciment No.</u>	<u>For</u>	<u>Uranium Particle Size</u>	<u>Percent of U density</u>
23-H	Control	840 to	61.3
25-H	Irradiation to 1000 MWD/T (2.5×10^{20} nvt)	420 μ	61.6
30-H	Irradiation to 3000 MWD/T (7.5×10^{20} nvt)		61.8
26-H	Irradiation to 3000 MWD/T (7.5×10^{20} nvt)	210 to	64.3
27-H	Control		62.0
28-H	Control	105 μ	64.6
42-H	Control	60 w/o 840 to	65.4
44-H	Irradiation to 3000 MWD/T (7.5×10^{20} nvt)	420 μ 40 w/o < 44 μ	67.1
45-H	Irradiation to 1000 MWD/T (2.5×10^{20} nvt)		66.3

Thorium-Uranium Alloy - J. W. Goffard

Three Th-2.1 w/o U-235 alloy specimens have been canned in zirconium capsules containing NaK as the heat transfer medium. They have been shipped to the MTR for irradiation to 1, 2, and 3 a/o burnup. The unrestrained dimensional stability of these specimens will be determined at the HAPO radiometallurgy facility after reaching the goal exposures.

The first of the six zirconium canned specimens previously sent to the MTR for exposure should be discharged at the end of this report period after attaining 1/2 a/o burnup. Reactivity and bend test data will be obtained on this specimen.

The Coaxial Tube Fuel Element Design - T. W. Evans

There is nothing new to report.

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Ceramics Laboratory - R. J. Anicetti, W. E. Roake

Placement of orders for equipment to fabricate ceramic fuel elements was essentially completed during the Quarter. Approximately 80% of the small capital equipment items have been received and installed. The extrusion press, the 50 ton general purpose press, the sintering furnace controls, and rubber mill are expected about May 1, 1956. Work was started on the electrical sub-station and hydrogen supply for the sintering furnaces.

UO₂ Fuel Materials - W. E. Roake

Examination of irradiated fuel pins containing UO₂ powders as described under this heading in previous reports awaits completion of rare gas analysis equipment.

Rupture of UO₂ Specimen in the MTR - W. E. Roake, D. L. Zimmerman

An UO₂ slug, 1.285" diameter and 4.00" long, sintered to 85 percent of theoretical density was jacketed in a 0.075" thick zircaloy-2 can. This slug failed on August 30, 1955, and released fission gas into the cooling water of the MTR Fuel Element Testing Facility. The details of the incident have been related in a previous report of this series (5).

After an exposure of about 2.5 MWD/T of UO₂ the fission gas was released through an obscure crack in a closure weld. The end cap and fractured section of weld metal has been removed from the slug and examined microscopically. The crack is observed to have characteristics of a hot, rather than a cold, crack. It seems probable that the crack occurred during welding of positioning lugs onto the jacket subsequent to can closure and autoclave testing.

The oxide slug was irradiated subject to a requested temperature limit of 2000 C in the hottest portion of the core. This was calculated to correspond to a conservative 22 KW/ft. for this slug. Subsequent events have indicated that the power generation during irradiation was higher than requested. The irradiated slug was cut in two normal to the long axis, one inch from the end of the oxide which operated at the highest temperature during irradiation. This point corresponds to the plane passing through the hottest point of the slug. This cross section is shown in Figure 4.15. No evidence of core melting was observed. The oxide is now loose in the jacket due to shrinkage by further sintering during irradiation. The oxide is cracked radially due to thermal stresses but not as badly as was expected. The coaxial tubular crack is indicative of radial stress. It can be assumed that the material within the zone bounded by the tubular crack shrank because of

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FIGURE 4.15 Cross Section of UO₂ Slug from GEH-4-3C. Photograph shows cross section of slug at hottest point. Cavity in center caused by broken piece falling out after sawing.

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sintering and thus was operating at temperature upwards of about 1600 C. Further effort is being made to conform the methods of temperature calculation with the experimental evidence. For this purpose a selected portion of the oxide will be analyzed for U²³⁵ burnup.

Uranium Reduction Studies - W. B. Tolley

Reduction experiments aimed at the preparation of uranium shot for use in the magnesium matrix fuel element have been discontinued. The particulate uranium metal can now be made by the Fernald personnel by a metal shooting process. The large bomb equipment for scale-up to 3000 to 4000 gram reductions has been received and will be installed as an autoclave for oxide and cermet fuel element testing.

Cermet Fuel Material Studies - W. B. Tolley

Little laboratory progress has been made on the cermet problem pending the installation of hydrogen atmosphere sintering equipment and arrival of metal powders for binder constituents.

Uranium carbides are being considered along with UO₂ and UO as base materials for cermet fuels. The monocarbide has a density of 13.66 g/cm³ and the dicarbide 10.52 g/cm³. Both are high melting materials. The fine powders do not ignite in air at temperatures below 380 to 400 C but are pyrophoric and can be ignited by crushing or grinding. A ten gram sample of the monocarbide has been prepared by the following reactions, and is being analyzed for carbon and uranium content:

If the product proves to be the monocarbide larger batches will be prepared for compacting and sintering studies.

Metal Coalescence Studies - W. B. Tolley

During the reduction studies aimed at preparation of uranium shot material and previous reduction studies of the uranium double salts and plutonium fluoride and chlorides, a considerable background in reduction processes has been accumulated in this laboratory (3, 4, 7, 8, 9, 10, 11, 12, 13, 14).

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Reductions of the oxides of the metals plutonium, cerium and uranium appear to be the more difficult systems from which to prepare useable metal. Results in this and other laboratories have been the same. Massive metal is not obtained. Pelleted or powder metal is obtained which is often pyrophoric, depending on the metal and variables of the reduction. The direct reduction of these metals via the oxide is an attractive goal which would considerably lower the present costs of converting a plutonium or uranium nitrate solution to the metal.

In reductions of the oxides and fluorides of uranium to yield massive metal it is usually necessary to take advantage of the exothermic nature of the reduction reaction, since temperatures above the melting point of uranium are necessary to form a solid button of metal. In general, construction materials do not permit operation at temperatures of 1200 to 1500 C unless special precautions are taken. However, plutonium which melts at 632 C permits operation well above its melting point using normal steel construction materials. Therefore, charges can be heated externally to the temperatures needed without dependence on the heat of reaction within the bomb to raise the charge to the desired temperature. Operation at these lower temperatures also allows one to soak the charges above the melting point of the metal and slag for long periods of time. For these reasons it was felt that the reduction of plutonium dioxide with calcium might be performed in a manner which would yield a solid metal piece. Since plutonium facilities were not available, cerium dioxide was used as a stand-in material. The melting point of cerium is essentially identical to that of plutonium; however, the densities differ by a factor of 2.75. It is felt that cerium is a fair stand-in for plutonium in these reduction experiments. Actually, as shown later in this report, the cerium probably results in poorer yields than can be expected with plutonium.

Previous work in this laboratory on the reduction of cerium dioxide with calcium has resulted in very poor metal button yields. Generally, powder or pellet material is obtained which is often pyrophoric. The following equation shows the products of the reduction reaction:

When no flux material is added to the charge, the high melting calcium oxide product forms a solid matrix preventing the cerium fines from coalescing. In this case extremely fine metal powder is obtained. Numerous experiments have been run using a flux material which partially dissolves the calcium oxide present resulting in a fluid slag. Again, pyrophoric powders are generally obtained. However, the metal is usually not as fine as that obtained without a flux. When enough flux is added to a charge to completely dissolve the CaO formed from reduction, and the charge is heated above the melting point of the metal and slag for 20 to 40 minutes,

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some solid metal is obtained. Yields have been low, 10 - 30 percent, and some fine powders are also recovered. However, more of the large pellets which can be handled in air are found in the slag. It was also noted that much of the cerium fines did not settle to the bottom of the charge as expected and that many fairly large sized pellets which had settled out had not coalesced. This was true of 1/16 inch diameter pellets which were side by side. It has long been realized that a certain amount of oxide present in the slags tended to hinder coalescence. In the reduction of PuF_4 and PuF_3 with calcium approximately ten percent PuO_2 in the compounds would result in poor button yields. The metal generally was somewhat spongy and often pyrophoric. It was also realized that the calcium oxide from the CeO_2 reductions was in fact the troublesome component in the coalescence difficulties. However, this was quite vividly shown in some recent work by W. R. DeHollander of the Metallurgy Research Sub-Section (15). He has shown that small additions of calcium oxide to slag systems from which metal had previously been coalesced would actually prevent complete coalescence of the metal under identical conditions. Apparently, a film layer of CaO around a molten metal pellet is sufficient to halt or at least slow the growth of the pellet in contact with other oxidized metal particles. But charges in which enough flux was added to theoretically dissolve all of the CaO formed during reduction still failed to give satisfactory metal yields even when soaked for 20 to 40 minutes at temperatures well above the melting points of both the slag and the metal. It is postulated that the kinetics of dissolution of calcium oxide in the calcium chloride flux are slow during the reduction. The undissolved calcium oxide then prevents good metal agglomeration.

A low frequency air-driven vibrator, 60 cycle/second, has been coupled to the reaction vessel during the reduction. It was theorized that this vibratory energy may markedly increase the dissolution rate of the calcium oxide in calcium chloride, and secondly, the film layers of undissolved calcium oxide may be physically disturbed, allowing metal to contact metal and coalesce. Using calcium chloride as the flux, quite impressive results were obtained from reductions of cerium dioxide. A mole ratio of $\text{CaCl}_2/\text{CeO}_2$ of 12.5 was employed in most reductions. This corresponds to an eight percent solution of calcium oxide in the calcium chloride slag after reduction has taken place. The solubility of calcium oxide in calcium chloride has been reported as 13.8 percent at 900 C so this is a large excess over the calcium chloride required for oxide dissolution. (12) The cerium dioxide, 100 percent theoretical excess of calcium metal and calcium chloride flux were mixed and packed in a fired magnesia crucible which was hermetically sealed in a steel bomb. The bomb was heated by induction to 1000-1050 C for periods of 20 to 40 minutes. During this heating period the bomb was vibrated for approximately two minutes at a time, these vibrations being repeated from five to ten times during the run. Cerium buttons of 40 to 50 percent theoretical yields were obtained. The unvibrated charges produced buttons of 10 to 30 percent yield. These vibrated yields represent an increase of 33 to 67 percent over the best yield obtained

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without vibrating the bomb. The bomb charge was heated to 600 - 700 C while vented, allowing a small amount of water vapor to be removed from the anhydrous calcium chloride. The bomb was then hermetically sealed with a pressure gauge. Pressures were negligible, two to five psi, if this procedure was followed. The low calcium vapor pressure prevents a rapid reduction reaction. In these diluted charges the cerium dioxide and liquid calcium may not come in contact for reaction to occur. If there were an appreciable calcium vapor pressure, the reaction would be expected to go to completion much more rapidly. It is therefore felt that less than 100 percent yields may in part be due to incomplete reduction of the cerium dioxide. As previously mentioned, the difference in the density of cerium and plutonium may play an important role in the effective metal coalescence. In the cerium system the metal to slag density ratio is approximately 3.15, while in the plutonium system the ratio is 8.67. It is expected that better yields will be realized from the reduction of plutonium dioxide than have been obtained from the cerium system because of this density difference.

A second method of performing the oxide reduction has been briefly studied. If a lesser amount of calcium chloride flux is used, the reduction goes essentially to completion. However, the metal does not coalesce due to the undissolved calcium oxide present even though the slag may be quite fluid. It has been found that the cerium pellets can be increased in size by vibrating the bombs heated to a temperature of 1000 C. By this procedure metal particles can be grown to shot size which are not pyrophoric. The slag can then be easily dissolved and the metal particles coalesced under calcium chloride containing a small amount of calcium to reduce any oxide skin present (14). With cerium dioxide, yields of 50 - 60 percent solid metal have been obtained. This operation is, of course, less desirable than a single cycle process. However cerium particles obtained by this method were large enough to be handled in air. If necessary, an inert atmosphere could be employed during the casting. During the slag dissolution step some of the cerium fines were oxidized by the aqueous solution and lost. Plutonium should be oxidized less rapidly than cerium, resulting in higher yields. However, some fines are always formed which will be partially oxidized during dissolution of the slag in water or dilute acids. These powders are often quite pyrophoric and would therefore cause difficulties if collected. In the case of cerium the fines were estimated to be 10 to 30 percent of the metal formed depending on the variables of the reduction. Due to the density differences the plutonium fines may settle to the bottom of the charge more readily than cerium. In such a case they may be more rapidly grown to a size which can be handled, resulting in a greater button yield.

The results with cerium dioxide have been encouraging. It is recommended that studies with plutonium dioxide be initiated. Several methods of dislodging the oxide skins could be studied. A range of frequencies from

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the low, 60 cycles/second through ultrasonics could be coupled to the reduction bombs. Jolting tables may find application and should be considered along with high temperature centrifugal casting equipment. The choice of a slag system may result in improved yields. The calcium chloride has been used since it could be easily leached away revealing the position, size, and shapes of the uncoalesced metal. However, several low melting fluoride slag systems thermodynamically compatible in a plutonium system might prove superior to calcium chloride (17,18). The use of magnesium metal as reductant may be a practical method of eliminating the incomplete reductions obtained in the dilute systems. Magnesium metal would have an appreciable vapor pressure at 1000 C approximately 280 mm of mercury. The reaction would then be a solid gas reaction instead of the liquid solid reaction which takes place with calcium and CeO_2 . Due to the solubility of magnesium vapor in the slag, the reduction reaction should proceed rapidly to completion. Although it is impossible to predict the degree of success which may be obtained from the plutonium oxide reductions, results with cerium indicate some work should be encouraged. In fact if quality button yields of 70 to 80 percent could be obtained with plutonium, a process may be economically feasible based on the recovery of the uncoalesced metal fines and unreduced plutonium oxide as a recycling feed material for subsequent bomb reductions.

Fission Product Studies - T. W. Evans

There is nothing new to report.

The Corrosion of Selected Reactor Materials in Dowtherm "A" - J. W. Goffard

The use of organics as reactor moderator-coolant has been considered for some time. Among the characteristics of certain organics that permit their consideration are low chemical reactivity, low induced radioactivity, good moderating capability, and low vapor pressure at elevated temperatures. The major disadvantage of the demonstrated high burnup fuel, U-Mg matrix, is the chemical reactivity that both constituents display toward water, the conventional coolant. A compatible behavior between an organic and U-Mg fuel material might promote the application of both to reactor technology.

A preliminary static corrosion test has been conducted to determine the behavior between one of the candidate organics, Dowtherm "A" and U-Mg fuel material and other associated reactor materials. The Dowtherm "A" (eutectic of diphenyl and diphenyl oxide, 26.5% and 73.5%, respectively) gradually darkened and the viscosity increased slightly during the test, but this was expected and was merely the result of pyrolysis. The test materials (Al, Zr, U-Mg, U shot, and Mg) remained bright and clean for

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200 hours and then slowly accumulated a dark coating. After exposure for 800 hours in the boiling organic (255 C), there was little evidence of corrosive attack. The most significant effect observed was the fouling or the formation of the carbonaceous matter on the metallic surface. Figure 4.16 shows the appearance of the test specimens before and after the 800-hour test. A more detailed discussion may be found in HW-41861, "The Corrosion of Selected Reactor Materials in Dowtherm 'A'", March 8, 1956, CONFIDENTIAL.

High Thermal Conductivity Casements for Thermocouples - F. B. Quinlan

Measuring the temperature of an in-pile test, whether it be a fuel element or not, has always presented numerous and difficult problems. Some work for the "Gamma Heating" tests at MTR led to the development of a sound pressure tight enclosure for thermocouples. In turn, this enclosure may be fastened to an especially prepared slug cap or test piece.

The technique consists of choosing a metallic tube of the correct size and material for the job, inserting a thermocouple lead through this tube, and then swaging the wall of this tube down onto the couple bead. The swaging is performed by an especially designed swaging die that draws the tube metal out into a solid, cold welded, metal point. The couple becomes embedded in this metal and enclosure and therefore has excellent thermal contact with the casing. Such tubes have successfully withstood pressure tests of 90 psi.

To date, aluminum and copper tubes have been swaged. Stainless steel tubing will be swaged in this manner as soon as harder steel dies are available. Joining of concentric tubes with a complete end closure has also been accomplished. Radiographs of some of these casements and end closures are shown in Figure 4.17.

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A.

B.

FIGURE 4.16

A. The Test Specimen before Exposure to the Boiling Dowtherm "A"

B. The Appearance of the Specimen after 805 Hours in Boiling Dowtherm "A". (1-1/3X)

Photograph Unclassified

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FIGURE 4.17 Plate I, No. 4 - Concentric 2S aluminum tubes
1/4 and 1/8 inch OD
No. 3 - 1/4 inch OD 2S Al tube, 20-mil
wall
No. 2 - 1/8 inch OD 2S Al tube.
No. 1 - 1/8 inch OD 2S Al tube, with
glass fiber insulated chromel
alumel thermocouple.

Photograph Unclassified

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(UNCLASSIFIED).

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RADIOMETALLURGY - L. D. Turner

Low Exposure Failures - D. P. O'Keefe

There was no work done on this study during the quarter ending March 31st.

H-Loop Fuel Elements Pertaining to Rupture Incident - G. R. Mallett

Pile Materials Sub-Section has requested the examination of two eight-inch slugs with an exposure of 360 MWD/T, one up-stream and the other downstream to a rupture that occurred in the H-Loop. They also requested the examination of a piece of the aluminum can wall from the rupture. The upstream slug had a corroded area and a hole on the top side of the slug. There is no measurable warp in this slug. The downstream slug has a double-throw warp of 0.062 inches with numerous pits on the convex side which is the side opposite the rib marks.

The piece of can wall, Figure 5.1, from the rupture has been metallographically examined. An X-ray diffraction pattern of both sides of the can wall was measured. Aluminum and alpha aluminum oxide monohydrate were found on the outside, and aluminum and some AlSi on the inside. The aluminum can wall was cross-sectioned for metallographic study. The outside of the can wall has extensive inter-granular corrosion, Figure 5.2, the inside has AlSi and the intermetallic layer, Figure 5.3.

The material that is in the corroded area of the upstream slug is brittle and flaky. Some of this material was removed and examined by X-ray diffraction, which showed it to be UAl_3 . A wafer was removed from the edge of this corroded area and one from the center. The uranium diameter on both of the wafers was measured to find if any uranium was lost since the uranium was exposed to pile water. The diameters of the wafer from the edge of the corroded area (5) are 1.330 inches from between the rib marks to the corroded area and 1.335 inches at 90° to this. The center wafer has an uranium diameter of 1.339 inches from between the rib marks to the corroded area and 1.338 inches at 90° to this.

The wafer from the edge of the corroded area has been polished and etched. The as-polished structure of the aluminum shows the aluminum on each side of the corroded area to contain Al-Si eutectic in the can wall, Figure 5.4, and severe intergranular corrosion, Figure 5.5, on the surface. As the bond layer approaches the corroded area the AlSi becomes gradually consumed until no AlSi can be detected and the intermetallic layer has become thicker until it is about 0.015 inches thick. The etched macro-structure reveals two distinct areas as shown in Figure 5.6. The area under the corroded can wall is darker in appearance and is outlined by columnar grains. This indicates that this area was in the uranium beta temperature phase. The remaining part of the structure appears

(5) All footnotes are referenced at the end of this section.

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FIGURE 5.1 Piece of aluminum can wall from the rupture that occurred in the H-Loop. Outer surface. 1X

FIGURE 5.2 Outer surface of aluminum can wall from the H-Loop rupture showing intergranular corrosion. Polished and etched for 2 minutes in 0.5% HF. 200X

Photographs Unclassified

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FIGURE 5.3 Inside surface of aluminum can wall from the H-loop rupture showing the AlSi and inter-metallic layer. Polished and etched for 1 minute in 0.5% HF. 200X

Photograph Unclassified

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FIGURE 5.4 The as-polished structure of the aluminum can wall adjacent to the corroded area. Note the AlSi eutectic adjacent to the intergranular corrosion of Figure 5.5. 300X

Photograph Unclassified

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FIGURE 5.5 The as-polished structure of the aluminum can wall adjacent to the corroded area. Note the intergranular corrosion on the edge of the can wall. 300X

Photograph Unclassified

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FIGURE 5.6 Macrostructure of the wafer from the edge of the corroded area. The rib marks are on the lower portions of the picture. Vacuum cathodically etched. 3X

Photograph Unclassified

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for irradiated uranium. The microstructure of the area under the corroded can wall has very few twins, and many microcracks. The material here appears as if it had been well annealed. The remaining part of the microstructure is heavily twinned, so it must be, or have been, under extreme stress.

The as-polished condition of the wafer from the center of the corroded area shows a large crack, Figure 5.7, propagating from the edge at the corroded area towards the center. The structure of the can wall and bond on each side of the corroded area is similar to that shown with the center wafer from this slug. This wafer will be examined metallographically.

The downstream slug has had a wafer removed from its center and will also be examined.

Zirconium Process Tube Rupture - W. S. Kelly

A slug rupture which occurred in the H-Loop at 100-H resulted also in a rupture in the zirconium process tube. Sections of the tube were removed and shipped to Radiometallurgy for examination.

Examination of the tube sections showed the rupture to be a hole almost rectangular in shape, with dimensions of approximately $3/8'' \times 1/2''$. The edges of the break were sharp, with no stretching or visible necking down. Cracks extended out about $1/4''$ from the corners of the hole.

The tube rupture occurred approximately 210'' from the rear Van Stone flange and was adjacent to the slug rupture.* Samples were available from this region and from sections taken within 8'', 169'' and 500'' from the rear Van Stone flange. Rockwell hardness values of the samples ranged from 54 RA at the rupture to 62 RA 40 inches downstream from the point of rupture. The hardness of a unirradiated control sample was 55 RA.

Samples from each piece are being analyzed by the Analytical Laboratories Unit. Preliminary results showed the hydrogen content to be slightly high, which could account for the hardness values shown above. The oxygen and nitrogen contents were normal.

* Personal communication with A.G. Blazewitz.

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FIGURE 5.7 Photograph of the as-polished wafer removed from the center of the corroded area. The rib marks are on the lower portion of the picture. 1.2X

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Examination and Sampling of Slugs from Project Bluenose - C. L. Boyd

Examination and sampling of selected slugs from Run V, Bluenose irradiation, ⁽¹⁾ has been requested by the Chemical Unit to accurately determine the radial distribution of fission products and correlate these results with those obtained by scintillation scanning.

Grinding equipment has been designed for selective removal of 0.002 - 0.003 inch thick samples from a 1/4 inch square bar sectioned transversely across an irradiated slug diameter. Preliminary results indicate that good recovery of the uranium from the silicon carbide grinding material is possible.

Three selected slugs (Numbers 3, 19, and 34 from the front face of the pile) have been measured, photographed, and wafers sectioned and scanned on the gamma scintillation scanning equipment. Results of the scanning operation indicate that slug 34 from the front face has a symmetrical distribution of fission products. This slug has been selected for additional sectioning to provide a 1/4 inch square transverse bar for selective sampling.

Examination of Warped Slugs from PT-313-10-7M - C. L. Boyd

Two warped, beta heat treated, lead dip canned slugs from PT-7M have been selected for examination to determine whether hardness, flux symmetry, and orientation may be related to warp. A description of the irradiation conditions was published in a previous report of this series. ⁽²⁾

A wafer from the central portion of each slug was scanned with the gamma scintillometer to determine the relative concentrations of fission products across this transverse section of the slug. The fission product concentrations as measured by the (0.75 mev) gamma activity are proportional to the integrated power or exposure of the small volume of material sampled by this measuring device. Hence the relative specific power generation across the slug or flux environment during irradiation can be ascertained. The degree of flux asymmetry found in the slug with 0.010 inches warp was similar to that in the slug with 0.088 inches warp. Thus, of the several factors that are usually regarded as contributing to warp in HAPO fuel elements; namely, residual stresses, irregular grain orientation, and abnormal heating caused by water flow irregularities or flux asymmetry, it appears that no correlation can be made between the magnitude of flux asymmetry and degree of warp. Rockwell hardness measurements on the slug with 0.088 inches warp varied from 96-97 R_G scale at the outer edge to 89-90 R_G at the center. No variation in hardness was observed between the concave

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and convex side of the warped slug. The other slug, which had 0.010 inches warp, varied from 96-97 to 92-93 R_G scale at the outer edge and center respectively. X-ray data has been obtained on a wafer from each slug, but the data has not been interpreted.

Examination of Cap-Type Slug Failures - D. L. Zimmerman

An increase in the number of cap-type slug failures during recent months prompted a request by Fuel Technology personnel for detailed examination of several selected ruptured and unruptured production slugs. One rupture and three non-ruptures have been examined to date and are listed in Table 5.1.

TABLE 5.1

Exposure Data of Examined Slugs

<u>Slug No.</u>	<u>Description</u>	<u>Tube No.</u>	<u>Exposure</u>	
			<u>MWD/T</u>	<u>NVT x 10²⁰</u>
F-11	8 ⁰⁰ Non-rupture	3574-DR	720	2.03
G-3137	8 ⁰⁰ Non-rupture	1185-H	754	2.13
F-3555	8 ⁰⁰ Non-rupture	4164-H	659	1.86
F-27	8 ⁰⁰ Cap Failure	4274-H	622	1.76

All of the unruptured slugs were selected because of a circumferential necked-down region in the aluminum jacket approximately 1/2 inch below the cap end. The severity of this necked region varied from a very noticeable distortion of the metal, as shown in Figure 5.8 to a barely perceptible band in which the metal distortion was evidenced by the flaking off of film deposits. In addition to the severe necking of Slug F-11, a pronounced elliptical shaped "hot-spot" was located at the cap end diametrically opposite the rib marks and was presumably formed by slug cocking. A portion of the hot spot can be seen in Figure 5.9.

The jacket of the ruptured slug (No. F-27) was split apart just below the cap core interface for approximately 2/3 of its circumference. After removal of the cap a crater approximately 1/4⁰⁰ deep was observed in the central portion of the uranium core. The peripheral area was relatively unattacked by pile coolant.

Length, diameter, and end concavity (dish) measurements were performed on the slugs as received and also on the uranium cores. These data are summarized in Tables 5.2, 5.3, 5.4, and 5.5.

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TABLE 5.2

Length and Dish Measurements - As Received

<u>Slug</u>	<u>Cap</u>	<u>Base</u>	<u>Length - Inches</u>		<u>Pre-Irrad.</u>
			<u>Axial</u>	<u>Peripheral - Avg.</u>	
F-11	*	22	8.799	8.827	8.80-8.94
G-3137	15	19	8.778	8.812	8.80-8.94
F-3555	29	15	8.749	8.793	8.80-8.94
F-27	*	*	8.904	8.905	8.80-8.94

* Too distorted for measurement.

TABLE 5.3

Diameter Measurements - As Received

<u>Position</u>	<u>Ave. Diameter Change from Pre-Irrad. Value-Mils</u>			
	<u>F-11</u>	<u>G-3137</u>	<u>F-3555</u>	<u>F-27</u>
Necked region	-12	0	-11	--
1" from Cap End	+17	+11	+18	+14
Center	+12	+ 8	+14	+10
1" from Base End	+17	+14	+17	+11

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FIGURE 5.8 Cap end of unruptured slug F-11

FIGURE 5.9 Longitudinal section of cap end of Slug F-11

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TABLE 5.4

Length and Dish Measurements - Uranium Core

Slug	Dish-Mils		Length-Inches			Length Change-Mils	
	Cap	Base	Axial	Peripheral-Avg.	Pre-Irrad.	L _A	L _P
F-11	16	29	8.251	8.296	8.325 ± .010	-74	-29
G-3137	8	21	8.250	8.278	"	-75	-47
F-3555	0	16	8.243	8.259	"	-82	-66
F-27	Corroded	30	--	8.354	"	--	+29

TABLE 5.5

Diameter Measurements - Uranium Core

Position	Avg. Diameter Change from Pre-Irrad. Value - Mils	
	F-11	F-3555
0.05" - 0.10" from Cap end of U	-13	-25
1/4" from Cap end of U	+ 6	-12
3/4" from Cap end of U	+11	+ 8
Center	+ 3	+ 4
3/4" from base end of U	+ 8	+ 6
1/4" from base end of U	+13	+ 9
1/8" from base end of U	- 7	-12

Both ends of slugs F-11 and F-3555 were sectioned longitudinally. Examination of the cap section of slug F-11 (see Figure 5.9) revealed that the uranium-AlSi bond below the cap had fractured as a result of the uranium core dishing. It was also observed that the jacket was separated from the uranium for a distance of approximately 1/8 inch from the end of the uranium. This separation was observed to end slightly above the area of most severe necking. The base end of this slug was dished considerably more than the cap end, however, there was no evidence of separation. The thinner section of the base presumably

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allowed it to conform to the uranium distortion without breaking the Al-Si bond.

The base-end of slug F-3555 was found to be slightly dished and the cap end perfectly flat with no evidence of separation. A very pronounced longitudinal crack was observed in the uranium cores of each of the sections. These cracks extended along the axis for the entire length (1") of each section.

Corrosion of Slugs Between Ribs - G. R. Mallett

Pile coolant Studies Unit has requested the examination of three eight-inch ruptured slugs. The type of corrosion between the ribs and the rupture mechanism are being determined. These slugs are from tubes 3173-F, 1581-H and 1381-H and have exposures from 510 to 890 MWD/T.

Each slug has a one-half to three-fourths inch hole between the rib marks one to two inches from the cap end. The area between the rib marks, of the slugs from tubes 3173-F and 1581-H, is extremely rough and recessed with respect to the rib marks, Figure 5.10. The slug from tube 1381-H, although recessed between the rib marks is smooth in this area. What appeared to be a scale on the slug from tube 3173-F was scraped off and analyzed as alpha aluminum oxide monohydrate by powder X-ray diffraction technique.

Wafers measuring one-half inch thick were removed from each slug. The slugs were sectioned through the center of their holes. The slug of tube 3173-F had another wafer removed from it, four inches toward the base end from the hole. The hole in the slug of tube 3173-F is one-half inch deep, Figure 5.11. The other two slugs have holes that are one-fourth of an inch deep.

Can wall thickness measurements have been made on both of the wafers cut from the slug of tube 3173-F. Adjacent to the hole, the can wall thickness was very thin, but gradually increased to 0.034" thick at 90° to the hole and 0.040" at 180° from the hole. The AlSi is 0.010 to 0.020 inches thick all the way around.

Further study of these wafers is underway.

The Examination of Cored and Solid AlSi Bonded Slugs Irradiated in the MTR (GEH-4-1) - C. L. Boyd - T. W. Evans

The first group of slug specimens to be irradiated in the HAPO fuel element testing facility in the MTR consisted of a standard AlSi canned solid slug, an AlSi canned cored slug (3), and a nickel-plated hot-pressed solid slug (4).

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FIGURE 5.10 Eight-inch rupture from Tube 1581-H with an exposure of 520 MWD/T.

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FIGURE 5.11 The half-inch wafer from Tube 3173-F, sectioned through center of the hole. As cut surface.
0.97X

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The purpose of this irradiation was to determine the behavior of a standard HAPO fuel element and two candidate replacement elements in the environment of the testing facility. A description of the testing facility, irradiation conditions, and previous post-irradiation examination results may be found in earlier reports of this series. (2, 3, 5)

A wafer from the central region of the AlSi bonded solid slug was scanned with the scintillation scanning equipment (6) to determine the relative concentrations of fission products across this transverse section of the slug. The fission product concentrations as measured by the (0.75 mev) gamma activity are proportional to the integrated power or exposure of the small volume of material sampled by this measuring device. Thus the relative specific power generation across the slug or the flux environment during irradiation can be ascertained. Results of the scintillation scanning showed a highly asymmetrical radial fission product distribution similar to that previously reported (2) for the cored slug. The concentration of fission products at one edge of the slug was 50 per cent greater than that found at the opposite edge. No warp or other dimensional instability was associated with this highly asymmetrical flux distribution.

Rockwell hardness measurements obtained on the wafer from the AlSi bonded solid slug varied uniformly from 96-98 R_G scale to 89-91 R_G scale at the outer edge and center respectively. These values indicate that no detrimental or asymmetrical heating effects were associated with the asymmetrical flux distribution. Conversely, hardness results obtained from the cored AlSi bonded slug wafer showed a noticeable variation in hardness. Hardness values across the region of lowest flux density decreased from 94-97 R_G scale at the edge to 89-91 R_G scale at the inner surface of the core, as compared with a decrease from 99-101 R_G scale to 79-82 R_G scale across the region of highest flux density. Additional metallographic work is planned to attempt to determine whether the observed hardness variation across the cored slug may be associated with abnormal heat transfer conditions set up by cracking of the uranium core or abnormal heating caused by the highly asymmetrical flux environment.

Internally and Externally Cooled Wafer Slug Failure - W. S. Kelly

Three internally and externally cooled aluminum jacketed uranium wafer slugs four and one-half inches in length were irradiated in the MTR to an estimated 700 - 900 MWD/T (2×10^{20} nvt) at the request of the Fuel Element Development Unit. Each slug had a different pre-irradiation canning history. The jackets of two of the slugs were AlSi bonded. One of the AlSi bonded slugs had the aluminum spacers separating the 0.125 inch thick uranium wafers AlSi bonded to the wafers. The four mil aluminum spacers in the second AlSi bonded slug were separated from the uranium wafers by one mil thick anodized aluminum foil to prevent

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bonding. The third slug was a nickel plated hot-pressed slug, with the six mil aluminum spacers covered with a one mil anodized aluminum foil ostensibly to prevent bonding of the spacers to the uranium wafers.

The slugs were discharged when a water tube monitor indicated a rupture. Examination of the slugs at the MTR showed the hot-pressed slug to be the only slug with any visible signs of rupture. It was discovered upon the arrival of the slugs at the Radiometallurgy Building that the severity of the rupture of the hot pressed slug had increased during the period the slug was in transit. Cracks which had been of a hairline nature immediately following discharge from the MTR had opened to about one-sixteenth inch in width. There was no visible deterioration of the AlSi bonded slugs.

A wafer was removed from each of the three slugs and used for Gamma scintillation studies. Results showed a maximum difference of 19% in Gamma radiation intensities at points equidistant from the wafer center but along different diameters.

Metallographic examination of sections of the hot pressed ruptured slug showed considerable intergranular corrosion of the inner can wall but none of the outer can wall. (Figure 5.12). The severest corrosion was at those regions where the can wall and aluminum spacers joined. Necking down of the can wall occurred at some of these same regions. Localized diffusion bonding was observed between the uranium wafers and the aluminum spacers at some of the points where the anodized layer on the foil had been broken by the canning operation, (Figure 5.13).

The preferential corrosion of the inner can wall is more understandable when it is noted that the calculated water temperature in the inner water passage was 170°C and that in the annulus between the process tube and the outer can wall 145° C during the period of irradiation. Therefore it is believed that the initial failure occurred in the inner water passage as a result of intergranular corrosion. The initial break, or breaks, allowed water to enter and cause the buildup of uranium oxide and hydride compounds on the surfaces of the uranium. This pushed the uranium wafers away from the aluminum spacers, stressing both the outer and inner can walls until transverse cracking occurred along the line of intersection of the spacers with the can.

The Effects of Irradiation on Uranium Bond Tensile Specimens - C. L. Boyd

Eight preformed tensile sample assemblies from a production test (7) which was designed to obtain information concerning the effect of irradiation on the properties of bonds obtained by the current hot-press slug canning techniques were received and opened. The tensile samples have a 1/2 inch long plated, beta heat treated, uranium mid-section diffusion bonded to

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FIGURE 5.12 Intergranular attack on the inner can wall of a Hot Pressed Internally and Externally cooled Wafer Slug. 25X

FIGURE 5.13 Aluminum-uranium diffusion bonding in a Hot Pressed Internally and Externally cooled wafer slug. Mag. 180X. Aluminum spacers and foil are white and uranium is light grey. The anodized layers on the foil show up as dark grey lines.

aluminum ends. The post-irradiation appearance of the uranium mid-section is shown in Figure 5.14. Half of the samples have a nickel-bonding layer and the others nickel-iron. Four of the tensile specimens were irradiated to a calculated exposure of 300 MWD/T and the remainder to 600 MWD/T. The irradiation temperature of all samples was 200°C.

Six of the irradiated specimens were in good condition with no obvious warping or deterioration of the bond. The other two samples, one with a nickel-iron bonding layer and the other with nickel, broke during opening or had deteriorated during irradiation. The nickel bonded specimen broke at the nickel-aluminum interface while the nickel-iron bonded sample parted partly at the nickel-iron-uranium interface and partly at the nickel-iron-aluminum interface.

Metallography of Irradiated Materials - James R. Morgan, Gavin R. Mallett and R. E. Field

The easy response of uranium microstructure to cold work during metallographic preparation warrants careful selection of polishing procedures to insure valid results. It was therefore decided that the first use of the Hanford-built remote polisher and cathodic vacuum etcher should be a study of the effects of operational variables on uranium microstructure. Such use on unirradiated samples seemed desirable not only to collect valuable data but also to permit easy correction of minor operational details of the new equipment before it became contaminated with highly radioactive material.

Six wafers of unirradiated uranium were cut from a normal Hanford fuel element and subjected to various metallographic polishing procedures. X-ray line broadening was used to measure the amount of cold work present on the surface at several stages in the course of remote mechanical polishing and remote cathodic vacuum etching. The (154) alpha line at a two-theta value of about 140° was used and its broadness expressed as the ratio of width at half height to peak height above background.

All the wafers were first annealed to remove residual cold work from slug fabrication and sectioning. Each wafer was then electropolished and X-rayed to establish a control value of line width in the annealed condition. Various mechanical polishes were given the wafers as shown in Table 5.6. Mechanical polishing and X-ray study was followed by a light cathodic vacuum etch before the surface had time to tarnish appreciably. X-ray lines were taken again after etching. The etched surface was repolished on the Linde A final stage just enough to obliterate the etch and cathodic vacuum etched a second time and X-rayed. The surface was again repolished lightly, cathodic vacuum etched a third time and X-rayed.

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FIGURE 5.14 Post-irradiation appearance of the uranium mid-section from one of the 300 MWD/T diffusion bonded tensile specimens.

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Results of the work completed to date are shown in Table 5.6. Future plans for this project included a study to determine if irradiation influences the sensitivity of uranium to work effects.

TABLE 5.6

X-Ray Line Broadening in Uranium during Metallographic Preparation

Wafer No.	1	2	3	4	5	6
Line width* as annealed	0.18	0.20	0.21	0.20	0.22	0.22
Minutes on 80 grit sandpaper	6	15	60	6	4	12
Minutes on 320 Gritcloth	15	15	60	7	Omit	7
Minutes on 600 Gritcloth	7	15	60	4	8	Omit
Minutes on Microcloth with Linde A in oxalic acid	7	15	60	60	6	3
Line width after polish.	**	**	0.47	**	**	**
Line width after light cathodic vac. etch. (30 min. 7 1/2 Ma)	1.10	1.27	0.29	--	1.60	--
Line width after light repolish on microcloth and Linde A and normal cath. vac. etch. (60 min. 7 1/2 Ma)	0.51	--	0.38	--	0.93	--
Line width after light repolish on microcloth and Linde A. Normal cath. vac. etch, (60 min. 7 1/2 M.)	0.40	--	0.27	--	--	--

* The expression line width as used here is defined as width of the (154) α_1 line at half height divided by the height of the peak above background.

** α_1 and α_2 lines not resolved.

Metallographic Cell - J. R. Morgan, G. R. Mallett, R. E. Field

The metallographic cell resumed normal operation on March 6th after having been shut down November 15, 1955 for a complete overhaul. New equipment installations included: (1) Hanford-built grinder-polisher,

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(2) filtering system for collection and disposal of polishing wastes, (3) remote cathodic etcher and vacuum system, (4) improved lighting, viewing, and macro-photography, (5) sample storage cave within the cell to reduce radiation levels at lenses and other equipment that is susceptible to radiation damage. Reusable equipment was renovated and returned to the cell. A general view of the cell exterior and interior is shown in Figure 5.15.

Testing of the new equipment began about February 1st. Some valuable data on the amount of cold work induced by metallographic preparation was obtained during the try-out period in addition to finding and correcting certain operational difficulties in the remotized, equipment. Results of the polishing and etching studies on unirradiated test pieces are reported in the preceding paragraph.

Remote Grinder-Polisher - R. E. Field, J. R. Morgan, G. R. Mallett

An additional grinder-polisher, to supplement the prototype unit within the metallographic cell, is now being fabricated. Certain modifications to the original design have been incorporated in this new unit such as use of ball bushings, aluminum sand casting for main housing, better vision of the sample and repositioning of hand controls to permit more efficient operation of unit.

Cathodic Vacuum Etching - J. R. Morgan, G. R. Mallett, R. E. Field

A new cathodic vacuum etcher was installed in the metallographic cell as was indicated in our last quarterly report (2). Tests with unirradiated and later with irradiated uranium indicated that the etcher functioned as well as the old model and was much easier to operate. Indications are that the new design, which has fewer and simpler moving parts within the cell, will give fewer maintenance problems and also will be easier to repair should difficulties develop.

The same sample of irradiated uranium (1.5×10^{20} nvt), used in tests on the old etcher was etched with the new unit. The microstructures as developed in the old and new etchers are compared in Figure 5.16 where no significant difference can be noticed.

Experiments designed to show the optimum procedures for both polishing and etching are being conducted as time permits.

Macro Positioner - R. E. Field, J. R. Morgan, G. R. Mallett

The macro positioner (8) was received and the entire assembly including camera, lead glass plug, positioner and control plug have been assembled in the cell mockup for testing. It is hoped that testing will be completed and the unit installed in the metallographic cell during the next two months.

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FIGURE 5.15 Exterior and Interior views of Hanford Metallographic Cell after overhaul and rearrangement of equipment.

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Comparison of Microstructure of irradiated uranium obtained with the old and revised cathodic vacuum etcher. Left: Etched in old etcher. Right: Etched in Revised etcher. Same wafer, (irradiated to 1.5×10^{20} nvt) used in both cases. Magnification: 425X.

FIGURE 5.16

Photographs Unclassified

SECRET

Project CG-660 Metallographic Cell Addition - R. E. Field, J. R. Morgan, G. R. Mallett

An extensive program of modifications to the metallographic cell including design and procurement of additional new equipment, was initiated and authorization granted for funds to scope the entire project and to purchase critically needed equipment immediately.

The proposed project will provide the following major modifications and additions to the metallographic cell:

1. Four grinder-polishers and drives
2. One additional vacuum cathodic etcher
3. Control panel and desk for etchers
4. Seven additional lead glass viewers
5. Ultrasonic generator and cleaning unit
6. Provide and equip cell extension for:
 - a. B and L research metallograph
 - b. Replication equipment
 - c. Electro-chemical etching unit

The proposed modifications are expected to increase the present examination rate in the metallographic cell from four per week to approximately fifteen per week. Maintenance and operation of equipment will be greatly simplified.

All design, procurement and installation of equipment will be made by the Project and Design Sections of the Engineering Department. Purchase requisitions have been initiated by these sections to procure the four grinder-polishers, cathodic etcher and lead glass viewers.

X-Ray Sample Holder - R. E. Field , W. V. Cummings

A sample holder for the double crystal X-ray diffraction unit was designed and fabricated. This holder was specifically designed to contain a minimum number of motions to permit exact adjustment of sample plane with respect to the X-ray unit. A combination of both rotation and radial in and out travel of the complete unit was used to meet this requirement. An additional motorized synchronous motion was also introduced to the sample holder to allow diametral scanning of samples at the rate of 0.10 inches per minute. This unit is very easily installed and aligned and due to its sturdy construction, should serve as an accurate standard for future studies.

1-Ton Transfer Cask - R. E. Field, W. S. Kelly

Performance of the recent 3-ton transfer cask has proven so successful, that a smaller cask of the same type has been designed and will soon be purchased off-site. This all stainless steel cask, which has a rotating

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cylinder to replace one end plug, will be used exclusively for sample transfer within the Radiometallurgy Building.

MTR Cask - R. E. Field, W. S. Kelly

The three-ton MTR transfer cask was received during the month of February. This stainless steel lead-filled cask was designed for shipment of preformed irradiated samples to the Radiometallurgy Laboratory. It will also be used for inter-area transfer of fuel elements between the Reactor areas and the Radiometallurgy Laboratory. The cask has an inner cavity six inches in diameter and eleven and one-half inches long which will permit three canned ruptured slugs to be transferred at once. By using a lead filled liner in the cavity the normal lead shielding of nine inches may be increased to eleven inches.

References

- (1) "A Compendium of Hanford Effort In Support of Project Bluenose," April 12, 1955, HW-36076 (SECRET).
- (2) Quarterly Progress Report, Metallurgy Research Sub-Section - Oct.-Dec. 1955, HW-40858, (SECRET).
- (3) Quarterly Progress Report, Metallurgy Research Sub-Section - Oct.-Dec. 1954, HW-34455, (SECRET).
- (4) Sprowl, J. D., and Bailey, W. J., "Fabrication and Characteristic of Hot-Pressed Fuel Elements," Proceedings of Spring Metallurgy Conference, 1954.
- (5) Quarterly Progress Report, Metallurgy Research Sub-Section - Jan.-Mar. 1955, HW-36054, (SECRET).
- (6) Paul, R. S., "A Method for Measuring Fission Distributions in Hanford-Type Fuel Elements," Jan. 24, 1956, (CONFIDENTIAL).
- (7) Johannesen, J. C., "Production Test No. 105-537 SI, Irradiation Service Request HAPO-165, Irradiation of Uranium Bond Tensile Specimens," Dec. 2, 1955, (HW-40263), (SECRET).
- (8) Quarterly Progress Report, Metallurgy Research Sub-Section, July-Sept. 1955, HW-39495, (SECRET).

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CORROSION AND WELDING - W. R. SmithCorrosion of Types 304-L and 347 Stainless Steel and A-55 Titanium by Oxalic Acid - W. L. Walker

In order to more fully investigate the corrosive characteristics of oxalic acid and to determine its suitability as a repetitive cleaning agent for stainless steel process equipment, a more detailed investigation than that described in HW-40858⁽¹⁾ was performed. Samples of types 304-L and 347 stainless steel which had been subjected to a sensitizing heat treatment were exposed to 10 w/o oxalic acid at temperatures from 25 to 101 C for time periods of from 0.5 to 24 hours. In addition to the stainless steel, samples of A-55 titanium were exposed to 10 w/o oxalic acid at temperatures of 25 to 101 C for periods of 2 and 100 hours. The results of these exposures are shown in Figures 6.1 and 6.2, and Table 6.1.

All the results are in good agreement with preliminary work done by this laboratory⁽²⁾ and work performed by other investigators; with the exception of the 50 C exposures of 304-L at 8 and 24 hours. These two points will be checked again before the final report is published.

The data shows that 10 w/o oxalic acid is a satisfactory repetitive cleaner for stainless steel process equipment, from a corrosion viewpoint, where corrosion rates on the order of 0.006 to 0.020 ipm can be tolerated for short periods of time. Oxalic acid should not be used as a cleaning agent for titanium process equipment unless large corrosion rates on the order of 0.100 ipm can be tolerated for short periods of time. A final report, HW-40804, detailing the investigation and results is being issued.

TABLE 6.1Titanium in 10 w/o Oxalic Acid

<u>Time (hrs.)</u>	<u>Temperature</u>	<u>Corrosion Rate*</u>
2	25°C	0.00000
100	25	0.00010
144	35	0.00005**
144	60	0.03750**
144	100	0.10353**
2	101	0.1939
72	101	0.1760

* inches penetration per month

** Lane, I. R., et. al. ⁽³⁾

(1, 2) All footnotes are referenced at the end of this section.

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FIGURE 6.1
CORROSION RATES OF TYPE 304L STAINLESS STEEL, WHICH
HAD BEEN HEAT TREATED AT 1250°F, 1 HOUR, AND WATER QUENCHED
IN 10 w/o OXALIC ACID

FIGURE 6.2

CORROSION RATES OF TYPE 347 STAINLESS STEEL, WHICH
HAD BEEN HEAT TREATED AT 1250°F, 1 HOUR, WATER QUENCHED
IN 10 w/o OXALIC ACID

Inhibition of Nitric Acid Corrosion - W. L. Walker

Work performed by other investigators⁽⁴⁾ has indicated that the addition of urea to nitric acid should result in an appreciable reduction in the corrosion rate of stainless steel exposed to this solution. However, previous work⁽¹⁾ performed by this laboratory has not agreed with their findings. Since the reduction of corrosion in the Purex acid fractionators is highly desirable, the testing techniques used by this laboratory were re-examined, and it is believed that the source of disagreement has been corrected.

Essentially all previous exposures of stainless steel to nitric acid containing urea have been the same. The nitric acid solutions were made up to specified concentrations of urea and the samples were exposed to these solutions for various periods of time at elevated temperatures: at the boiling point of the solution or higher. Under these conditions, there has been no conclusive evidence of reduction of corrosion by urea. However, the postulated inhibitory action of urea in nitric acid is the reduction of NO^- and NO_2^- to free nitrogen, and at the boiling point the thermal decomposition of nitric acid is appreciable, amounting to about 10 percent. With urea present in the solution this thermal decomposition would proceed rapidly until all the urea had been depleted and then would approach equilibrium conditions for the specific nitric acid solution.

With these thoughts in mind, the testing procedure was changed in the following manner. Solutions of 65 w/o nitric acid were made 0.2, 0.1, and 0.01M with respect to urea and placed in water cooled flasks. A portion of the solution from each cooled flask was placed in a separate flask and brought to a boil. Samples were placed in each of the three boiling flasks, and solution from the water cooled flasks was added continuously at a rate of about 2 ml per minute for a total of 240 hours. Weight loss measurements were performed every 48 hours. The material used in this investigation was type 304-L stainless steel which had been subjected to a sensitizing heat treatment, and it exhibited a Huey test corrosion rate of 0.0018 ipm. The corrosion rates observed for this material in the modified Huey test were as follows: 0.2M urea, 0.0009; 0.1M urea, 0.0007; and 0.01 M urea, 0.0004. These rates represent reductions in corrosion by factors of from 2 to 4. An interim report, HW-42100, is being issued which details the work performed in this investigation to date, and further work is planned wherein the effects of lower concentrations of urea and sparging with oxygen will be investigated.

Measurement of the Surface Temperature of a Heat Transfer Wafer -
M. C. Fraser, W. L. Walker

Laboratory corrosion testing techniques in general are not adequate for the corrosion engineer to predict accurately the service life of a vessel

(4) All footnotes are referenced at the end of this section.

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in the field. However, some evidence has been collected by this laboratory which indicates that the heat transfer corrosion test units⁽⁵⁾ do provide data which are significant with respect to the life of a heat exchanger tube bundle.

In spite of this, the heat transfer units do not represent the most desirable method for corrosion testing. Their greatest shortcoming is the length of time required to produce adequate data.

The investigation of the effect of heat flux on the corrosion of a heat transfer surface and the demonstration that this effect is small was described in HW-40858⁽¹⁾. The results of this investigation led to the belief that the corrosion rate of a heat transfer surface is a function of the surface temperature of the metal. If this assumption is correct, then the correlation between the results obtained from the factorial autoclave corrosion tests (FACT test)⁽⁵⁾ and from the heat transfer units can be established on a basis of surface temperature. The consequence of this development would be the elimination of the time bottleneck in the production of laboratory corrosion data relative to heat exchanger service.

Thus, a project has been initiated to study the factors which influence the corrosion rates established from identical materials exposed to identical media in FACT tests and in heat transfer tests. The objective is to determine if these two test methods have a correlation on the basis of metal surface temperature. The initial scope of this project is: 1) to establish the surface temperature of a heat transfer wafer at various combinations of solution and bulk metal temperature in the range of 100 to 170 C; 2) to establish comparative corrosion data from pressurized heat transfer units which permit operating conditions similar to both the heat transfer units and the FACT tests; and 3) to investigate other variables which may affect the corrosion rates obtained from the capsules such as pressure, geometry, time, etc.

Apparatus has been designed and constructed for the purpose of measuring the surface temperature of a heat transfer wafer. This apparatus is the same as a heat transfer unit with the exception that two platinum-platinum 13% rhodium thermocouples were peened onto the surface of the heat transfer wafer. The temperatures measured by these thermocouples should represent an average of the horizontal temperature gradient, at or very near to the metal surface, across a one-quarter inch and a three-quarter inch diameter area within the one inch diameter exposed area of the wafer.

A preliminary investigation has been made on the effect of bulk metal and solution temperature upon the surface temperature of a heat transfer wafer. The solution temperature was varied between 100 and 111C and

(5) All footnotes are referenced at the end of this section.

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the bulk metal temperature between 125 and 165 C.

The data obtained are presented in Table 6.2. The boiling points listed were established by arbitrary additions of aluminum nitrate nonohydrate to deionized water. These data show that differences in bulk metal and surface temperature of a heat transfer wafer may be as large as 40 C.

A comparison of the effect of temperature on the corrosion rates, of a heat of 347 stainless steel, obtained from the FACT units and the heat transfer units shows good agreement when the heat transfer data are plotted as a function of surface temperature instead of bulk metal temperature.

[REDACTED]

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TABLE 6.2COMPARISON OF INTERNAL AND SURFACE TEMPERATURES
UNDER HEAT TRANSFER CONDITIONS

<u>Boiling Point</u>	<u>Internal Temp.</u>	<u>Surface Temp.*</u>
100	122	107
	130	109
	137	109
	146	110
	149	109
103	132	113
	137	113
	149	114
	160	115
	165	120
105	135	119
	148	120
	155	123
	157	125
	156	124
108	138	122
	140	125
	150	127
	160	130
	160	131
111	139	126
	145	128
	155	132
	165	135
	169	134

*Note -- Average of four surface areas.

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Heat-Transfer-Corrosion Tests to Determine the Corrosivity of Synthetic 2WW Purex Waste Acid Concentrate -- M. C. Faser

A program is in progress to determine the corrosivity of boiling synthetic 2WW Purex waste acid concentrate by quantitative, semi-quantitative and qualitative methods upon wrought and cast types 304-L, 347, and 312 stainless steel and wrought type A-55 titanium under heat transfer conditions. The threefold purpose of this program is: 1) the determination of the existence and order of magnitude of the corrosion problem which may be expected in the Purex acid concentrators; 2) the collection of engineering data to facilitate the judicious selection of replacement or repair materials and method; and 3) the establishment of the effects of time and temperature on corrosion for operational data. The test equipment and solution being used are described in HW-34455. (5)

Type 312 stainless steel is an iron-chromium-nickel (nominally 62%, 29%, and 9% respectively) alloy which has excellent corrosion and heat resisting properties. More important for this project however, it is a stainless alloy whose composition produces a two phase, ferrite and austenite, structure. Observations made at this laboratory during the course of several years corrosion testing of various stainless steel alloys indicated that the severity of intergranular corrosion could be reduced by the presence of ferrite. The subsequent investigation of ferrite containing "austenitic" stainless steels resulted in the choice of type 312 as an alloy for corrosion investigation for two reasons. "Austenitic" alloys containing ferrite in amounts less than forty percent were susceptible to hot shortness during fabrication. The composition of other ferrite containing "austenitic" alloys was such that the necessary balance was difficult to maintain during melting or they contained molybdenum or copper, both of which were undesirable from the viewpoint of resistance to boiling nitric acid solutions under heat transfer conditions.

Data have been collected on the corrosion resistance of two heats of type 312 stainless steel and on one heat of type A-55 titanium during exposure to boiling synthetic Purex 2WW waste acid concentrate. The effect of time and temperature upon the corrosion rate was investigated in both cases. The test conditions employed are listed in Table 6.3 and the compositions of the two heats of 312 are tabulated in Table 6.4.

(5) All footnotes are referenced at the end of this section.

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TABLE 6.3

Heat Transfer corrosion test conditions employed for investigation of the Corrosion Resistance of Type 312 stainless steel and type A-55 Titanium to boiling synthetic 2WW.

<u>Metal Type</u>	<u>Heat No.</u>	<u>Time and Temperature</u>							<u>Heat Treatment</u>
312	58A1	Hrs. °C	1274 115°	1274 125°	1274 135°	1274 145°	1274 155°	1274 165°	Sensitized
312	58A1	Hrs. °C	66 145°	130 145°	275 145°	550 145°	1024 145°	2048 145°	As Received
312	45A1	Hrs. °C	66 145°	130 145°	256 145°	512 145°	1024 145°	2048 145°	Sensitized
A-55	M-438	Hrs. °C	334 115°		334 135°	334 145°		334 165°	As Received
A-55	M-438	Hrs. °C	48 145°		146 145°	194 145°		334 145°	As Received

TABLE 6.4

Composition of Two Heats of Type 312 Stainless Steel

<u>Heat No.</u>	<u>Cr. w/o</u>	<u>Ni. w/o</u>	<u>Mn. w/o</u>	<u>Si. w/o</u>	<u>P w/o</u>	<u>S w/o</u>	<u>C w/o</u>
DJ-58A1	28.9	8.43	1.72	0.51	0.007	0.013	0.09
DW-45A1	28.9	8.97	1.83	0.52	--	--	0.027

The data are presented in graphical form in Figures Number 6.3 and 6.4. Data, which were presented previously⁽⁶⁾, on specimens from a heat of type 347 stainless and also plotted on Figure 6.3 for comparative purposes.

Figure 6.3 shows that the magnitude of the corrosion rate of the 312 stainless is less by a factor of 1.5 to 2 than the 347 stainless throughout the temperature range of 115 C to 165 C. It also shows that the slopes of the temperature versus corrosion rate curves for 312 and 347 stainless are approximately equal. Figure 6.4 shows that the corrosion rate of these two heats of 312 stainless increases gradually with time of exposure, at an average metal temperature of 145°C, with no indication of becoming constant. Heat 45A1 shows greater corrosion resistance under these experimental conditions than Heat 58 A1. This behavior may be due to the difference in carbon content of these two heats. Based on these limited data, type 312 stainless appears to offer greater corrosion resistance than type 347 stainless for Purex waste acid concentrator heat exchanger service.

The data collected on type A55 titanium indicates corrosion rates of very low magnitude. For periods up to 334 hours with metal temperatures from 115 to 165°C the corrosion rates are less than 0.0005 ipm.

(6) All footnotes are referenced at the end of this section.

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FIGURE 6.3

TEMPERATURE vs CORROSION RATE DATA FOR TYPES
347 & 312 STAINLESS STEEL AND TYPE A-55 TITANIUM EXPOSED
TO BOILING SYNTHETIC 2_{WW} PUREX WASTE ACID CONCENTRATE FOR
2048, 1274, & 336 HOURS RESPECTIVELY

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Corrosion Rate (ipm $\times 10^{-4}$)

FIGURE 6.4

GRAPH OF TIME VERSUS CORROSION RATE DATA FOR TYPE
312 STAINLESS STEEL AND A-55 TITANIUM EXPOSED TO BOILING SYNTHETIC 2w
PUREX ACID CONCENTRATE AT 145°C

Corrosion Test Heat Exchangers -- N. D. Groves, K. M. Haws

To obtain a better understanding of the corrosion effects on materials of construction and welding alloys in the waste, acid and uranium concentrators in Purex plant, five corrosion test heat exchangers (see Figure 6.5) were placed in the F-6, F-11, H-4, J-8, and K-4 concentrator overflow chambers in the Purex facility. These units will be operated under the same conditions as the process heat exchangers. The test units are single tube calandria type heat exchangers consisting of a 2" schedule 40 stainless steel pipe shell, a 3/4" schedule 40 stainless steel pipe as an inner tube and supplementary steam inlet and condensate return lines. A sample rack containing coupons of candidate construction materials to be used as a basis for evaluating corrosion of non-heat transfer components of the concentrator were attached to the lower end of each test heat exchanger. Both the shell and tube components were fabricated from types 347 and 304-L stainless steel pipe cut into short lengths and joined with 308-L and 347 welding alloys (see Figure 6.6). The "G.E."⁽⁵⁾ joint design and tungsten inert gas welding process were used for all joinings. To produce representative cross welds on the shell surface short weld beads were laid transverse to the butt weld joints at 90° intervals around the circumference of the pipe. These cross welds were finished flush to the surface of the pipe except at the starting point and weld crater which were left in the as-deposited condition. Each sample rack contains corrosion coupons made from 308-L, 312 and 347 weld metal, 347 plate butt welded with 347 welding alloy, 304-L plate butt welded with 308-L welding alloy, 312 plate butt welded with 312 welding alloy, tantalum, and titanium. The test units will be run either to failure or until failure occurs in the process heat exchanger which is in parallel with the test unit. No further reporting on these units is planned until one is removed from service and examined.

Evaluation of Failure of Studs for Remote Connections in Purex Separations Plant Equipment - K. M. Haws

During the emergency replacement program of type 414 studs welded to the vessel flanges with type 420 studs held in place by a mechanical key in Purex equipment several of the type 420 studs experienced brittle fractures. Investigation of these failures revealed that two lots of type 420 stainless steel studs had been purchased for spare parts replacement and were used in the Purex vessels. One lot of type 420 studs were very hard (51-53 Rockwell C) and studs from this lot were the ones which failed with typical brittle fractures. The other lot of studs were softer (41-43 Rockwell C) and it was recommended that only this lot be used as replacements. Later three of these studs failed when they were hammered into alignment with the vessel flange.

(5) All footnotes are referenced at the end of this section.

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Fig. 6.5

CORROSION TEST HEAT EXCHANGER

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BASE
MATERIAL

WELDING
ALLOY

WELDING
ALLOY

BASE
MATERIAL

Fig. 6.6 BASE METAL AND WELDING ALLOY USED TO FABRICATE SHELL AND TUBE HEAT EXCHANGER

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At this time an additional 300 of the type 420 studs were on order. These studs were being fabricated by the vendor who supplied the studs which experienced brittle fractures and had high hardness values. A test program was initiated to determine if an alternate heat treatment would reduce the hardness and improve the ductility of these studs without deleterious effect on their other mechanical properties. Torsion impact and tensile specimens were machined from both the hard and soft lots of type 420 studs in the as received conditions. Specimens were also machined from the hard studs after tempering at 1000 and 1200 F. Included in the test for comparison purposes were specimens machined from original type 414 studs from Purex, type 416 bar stock which had been quench hardened and stress relieved, type 1020 carbon steel and an 18-8 stainless steel. Sections cut from studs of both lots were tempered at different temperatures (Table 6.5). No decrease in hardness of the studs was observed below tempering temperatures of 800°F, which indicates they were stress relieved and/or tempered at or just below this temperature. Available information from Inspection Sub-Section indicated the softer studs had been tempered at about 900°F and the hard studs at 700°F. Hardness values run on these studs substantiate this information. The 400 series martensitic chromium stainless steels exhibit secondary hardening characteristics when tempered in the 800 to 1100 F range and if tempered in this range yield low and erratic izod impact values. Vendors literature cautions against tempering in this range. To reduce the hardness and improve the ductility of the harder type 420 studs it would be necessary to temper this material above 1100 F to avoid the secondary hardening temperatures.

The results of this test program show that the studs tempered at 1200 F gave the best torsion impact values (Table 6.6) but had tensile and yield strengths less than one-half those obtained from similar studs in the as-received condition. The actual strength requirements for this application are unknown but it is considered that the strength of the studs tempered at 1200 F may be inadequate.

It is concluded that type 414 and 416 stainless steel are the better materials for this application.

TABLE 6.5

Material	Hardness Values - Rockwell C Scale			
	As-Received Condition	Tempered At 800°F	Tempered At 1000°F	Tempered At 1200°F
Hard type 420 studs	51-53	51-53	37-39	26-28
Softer type 420 studs	41-43	41-43	37-39	24-27

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TABLE 6.6

Material	Heat Treatment	Tensile Strength psi	0.2% Off- set Yield Strength psi	Elong- ation in 2" %	Reduction of Area %	Torsion Impact Values in Ft/Lbs.	Average Hardness Values Rockwell C Scale
Hard type 420 SS studs	As-received condition	253,600	244,300	6.5	25.3		53
		244,300*	181,200*	3.0*	6.4*		52
						80	53
						80*	52
						58*	48
Hard type 420 SS studs	Tempered at 1000°F Oil quenched	154,000	123,200	12.5	34.6		37
						85	35
						67*	37
Hard type 420 SS studs	Tempered at 1200°F Oil quenched	114,000	82,000	16.0	56.4		24
						101	26
						102	25
Softer type 420 SS studs	As-received condition	183,400	153,700	8.0	19.5		41
						48	42
						54	43
Type 414 SS studs out of Purex	As-received condition	204,900	149,600	11.5	61.7		43
						60	44
						54	43

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TABLE 6.6 (Cont.)

Material	Heat Treatment	Tensile Strength psi	0.2% Off-set Yield Strength psi	Elongation in 2" %	Reduction of Area %	Torsion Impact Values in Ft/Lbs.	Average Hardness Values Rockwell C Scale
Type 416 SS bar stock	Quench hardened from 1800°F and stress relieved at 600°F	190,800	140,800	11.5	57.2	51 52	43 44
18-8 stainless	Annealed					133 133	
1020 Carbon steel						104 104	

* Indicates the type 420 studs which were obtained from Todd in the unfinished condition. The other type 420 studs were selected at random from those used in the Purex facility.

NOTE: Each tensile and torsion impact specimen was machined from separate studs. It is not known if the studs were fabricated from the same piece of steel, consequently the small variations in mechanical properties could be attributed to variations in composition.

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Examination of Mercury Switches - N. D. Groves

The integrity of the primary alarm circuit of HAPO reactors depends upon the reliability of mercury switches which actuate the alarm circuit. The mercury switches are an integral part of the Panellit pressure monitoring gauges and are operated by a magnet attached to the dial of these gauges. The mercury switches (manufactured by the Mercoid Corporation) installed on the early HAPO reactors were a single pull, single throw model which excited an alarm when operated. To facilitate locating the source of an alarm, a coordinate light system was incorporated into the design of KE and KW reactors. This system required a single pull, double throw model mercury switch to actuate both the alarm and the coordinate light system. During the installation of the single pull, double throw models (manufactured by Hamlin, Inc.) in the "K" reactors, several mercury switches were found which contained faulty weld joints within the switch⁽⁷⁾. The examination of these faulty switches on the "K" reactors indicated a need for an improved flexure point or "hinge", and thus, the switches procured for Project CG-558 included a bearing and journal as a pivot point. After approximately two months' operation, the switches installed on Project CG-558 are becoming "sluggish" and "sticky", i. e., the force required to operate the switches is increasing with increased service life, and some of the switches fail to reset after the magnet is removed.

Twenty-three Hamlin mercury switches, five malfunctioning CG-558 switches, six CG-558 switches with "good" bearings, six new CG-558 replacement switches, and six "K" reactor replacement switches were disassembled and examined microscopically and macroscopically to determine the nature of the malfunctioning. The examinations of the malfunctioning CG-558 switches revealed: (1) the presence of mercury between the graphite bearing and nickel journal, (2) particulate graphite suspended in this mercury, and (3) extensive deterioration of the inner surface of the graphite bearing. It appears that the mercury was drawn into the annular space between the bearing and the journal by surface tension and/or capillary forces, and subsequently, the graphite binder was dissolved by the mercury thus causing particulate graphite to float in the mercury until a sludge was formed and impaired the action of the bearing and journal. The examinations of the "K" reactor replacement switches and the CG-558 replacement switches, which had stainless steel journals, revealed no mercury in the bearing-journal contact area and no deterioration of the bearing surface. The details of this examination have been reported. ⁽⁸⁾

(7, 8) All footnotes are referenced at the end of this section.

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Based on the above observations, it is recommended that, in order to prevent mercury from entering the annular area between the graphite bearing and the journal, the journal be made of stainless steel, which is not wet by mercury, in all future mercury switches.

Joining Zircalloy II Process Tubes - L. C. Lemon

The technology of Zircalloy II fabrication has not been developed to the extent that full length HAPO reactor process tubes can be extruded. Consequently, three 20-foot lengths of the extruded tubing were joined by welding to form an experimental process tube. The butt joints were accomplished by inserting a copper sizing plug which was equipped to distribute inert shielding gas to the inside of the joint as well as accomplish lineup and control the contour of the root of the weld bead. The joint design involved a 60° included angle bevel in the region of the longitudinal ribs with no joint preparation on the remainder of the circumference. Thus the joint was prepared such that the wall thickness was the same over the entire circumference, allowing a fusion weld by the tungsten inert gas process with argon as the shielding gas. Subsequent to the fusion pass, filler material was added to the beveled area of the longitudinal ribs to complete the weld.

This technique produced acceptable welds from the view point of visual and radiographic examination. Currently, test welds made in the same manner are undergoing corrosion tests and post-pile-exposure examination of the experimental tubes will serve as a final evaluation of this welding method.

Butt Welded Joints for Pipe - L. C. Lemon

During the past eighteen months a program of development and evaluation of several joint designs for butt welding both austenetic stainless steel and carbon steel pipe has been conducted to develop a reliable joint design which can be inspected visually for penetration characteristics. This study has centered around three weld joints: 1) the "G.E." joint, developed in this laboratory and embodying most of the advantages of several other joint designs ⁽¹⁾; 2) the EB consumeable insert joint which permits the addition of alloying elements to the weld ⁽⁶⁾; and 3) the standard bevel joint which is commonly used for this application ⁽⁶⁾. Earlier phases of this study have evaluated the integrity, joint preparation requirements, fit-up, weldability, speed, versatility and visual inspectability of the three joints mentioned. ⁽¹⁾

(1, 6) All footnotes are referenced at the end of this section.

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Form cutting tools have been developed to facilitate the preparation of pipe joints having the G.E. configuration. These tools, as shown in Figure 6.7 are standard lathe tools fabricated from tool steel with carballoy tip inserts. Tool design No. 1 is suitable for cutting joints on pipe having a wall thickness within the range of $1/4''$ to $3/4''$, and tool design No. 3 is suitable for wall thicknesses of $3/32''$ to $1/4''$. The significant difference between these two tools is the dimension of the face of the tool, i. e., thinner wall sections require smaller joints than the thicker walled material.

"Roto-Arc" Welding Process - L. C. Lemon

A concerted effort has been carried out at HAPO directed toward increased automation of the fuel element canning operations. In support of this effort, a welding process which is particularly amenable to automation has been developed and demonstrated. The "roto-arc" equipment consists of a water cooled, cylindrical molybdenum electrode held above the can closure within a shielding enclosure. The fusion weld is produced by moving the arc very rapidly from one point to the next on the electrode by applying a magnetic field across the weld joint thus welding the entire closure continuously without producing a weld crater. The rate of arc travel is fast enough to give the appearance of welding the entire joint instantaneously. In this manner a closure weld is produced on an aluminum can by equipment which contains no moving parts.

The fact that this process will produce welds free of craters using equipment with no moving parts has been demonstrated using a molybdenum electrode. However, in order to adapt this process to automatic equipment and to increase electrode service life, considerable effort is being directed toward developing a pure tungsten electrode to replace the molybdenum.

Inasmuch as wrought cylindrical tungsten is not available commercially, an attempt was made to utilize sintered tungsten as an electrode material. The physical characteristics of this sintered material were not sufficiently adequate to withstand the high stresses induced by the rapid thermal cycling encountered during its use as an electrode and severe cracking resulted.

Currently, an effort is being made to fabricate an electrode of wrought tungsten by inserting a series of one-eighth inch diameter tungsten rods into the end of a cylindrical, mild steel base and copper brazing them to the mild steel.

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Tool design No. 1 - to be used for cutting "GE" joints on pipe having a wall thickness of 1/4 inch to 3/4 inch.

T	3/16
W	1/2
L	1/2
D	5/32
BR	40° - 60°
SR	60° - 100°
X	1/2
Y	1/2
Z	3 1/2
SC	50° - 70°
FC	50° - 70°
ECEA	30°
SCEA	30°

Tool design No. 3 - To be used for cutting "GE" joints on pipe having a wall thickness of 3/32 inch to 1/4 inch

Fig. 6.7 CARBALLOY TIPPED TOOL FOR CUTTING "GE" PIPE JOINTS

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Evaluation of AC-DC Rectifier Welder - L. C. Lemon

In an effort to improve the safety of operation and the quality of welding at HAPO, an evaluation of a new type of welding machine has been conducted. The advantages of the AC-DC rectifier welder when compared to conventional welding machines are as follows:

1. The AC-DC rectifier welder produces alternating current and direct current both straight and reverse polarity while conventional machines only produce either alternating current or direct current.
2. The characteristics of the arc action are equal to or better than those produced by a transformer or motor generator machine.
3. Although the AC-DC rectifier welder does the same job as two conventional machines, it costs about the same either of the conventional types.
4. The AC-DC rectifier welder has an automatic cut-off mechanism which shuts the gas, water and welding current off after a pre-determined time following the breaking of the arc, thus enhancing economy of operation and providing an additional safety device to prevent accidental burns on the operator and damage to the work being done.
5. This machine contains only one moving part, a small cooling fan, which tends to minimize maintenance costs. The selenium rectifiers are guaranteed for five years operation and development tests indicate this may be a very low estimate of service life.
6. With the elimination of moving parts, the noise of operation has also been eliminated thus reducing the strain and fatigue of the operator.

With these facts in mind it is strongly recommended that all replacement and/or new welders obtained at HAPO be of the AC-DC rectifier type in order that full advantage may be taken of the factors noted above.

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