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QUARTERLY PROGRESS REPORT

METALLURGY UNIT

APRIL, MAY, JUNE, 1954

by

J. J. Cadwell

Applied Research Sub-Section
Technical Section
ENGINEERING DEPARTMENT

July 16, 1954

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SUMMARYPHYSICAL METALLURGYMechanical and Physical Properties of Uranium

Four irradiated tensile specimens were tested in the Radiometallurgy laboratory. The uranium was from a lot rolled at Simonds Saw and Steel Company, then beta heat treated by the triple dip process. The samples were irradiated to 310 MWD/AT. The corrected exposure was 620 MWD/T after compensating for the decrease in flux depression due to the specimen size. Two of the irradiated specimens were tested in the as-received condition while the other two were vacuum annealed at 400°C for 15 hours prior to testing. When the tensile properties of the unirradiated controls, irradiated, and irradiated-annealed specimens were compared, it was observed that no marked change in modulus of elasticity occurred, the 0.1 percent offset yield strength increased by a factor of about two for the irradiated and irradiated-annealed specimens, and the percent elongation decreased by a factor of 50 for the irradiated, and 35 for the irradiated-annealed specimens, compared to the controls. The marked change in mechanical properties on irradiation as well as the relatively small effect of the anneal, indicates that the "foreign atom" effect caused by fission product concentration and distribution is the controlling factor, and work-hardening type of damage is relatively insignificant.

Additional specimens have been fabricated for exposures of 75, 150, and 1200 MWD/AT. The method of canning was similar to that used for the 310 MWD/AT specimens with the exception that all rather than part of the specimens were coated with colloidal graphite and all specimen edges were rounded to facilitate post-irradiation disassembly. The canning technique has been modified to include the use of two caps so that a second weld can be made in case the first is rejected. This modification has reduced reject weld reassemblies fifty percent.

An analysis of the feasibility of NaK canning for PT 105-3N, Supplement A, has been made; results indicated that this procedure is not entirely feasible for Hanford irradiation at this time so the cold canning of test specimens has been continued.

Tensile and electrical resistivity specimens have been canned and autoclaved, and are awaiting scheduling in the pile, these specimens will be irradiated at 10^{16} to 3×10^{18} nvt at a maximum temperature of 50°C to determine the effects of low temperature irradiations on the properties of uranium.

Tensile and impact specimens will be canned with NaK in Zircaloy-2 capsules for MTR irradiation. Exposures of 600 MWD/T and 1500 MWD/T at 400°C have been selected. Final revisions in specimen and capsule size are being made based on the completed heat transfer calculations.

Structural Materials

The embrittlement of Zircaloy-2 is being investigated, using impact and stiffness testing methods. Addition of 200 ppm of hydrogen decreases the impact strength at room temperature by a factor of greater than two, and appears to change the fracture from predominantly transgranular in the hydrogen free to predominantly intergranular in the specimen containing 200 ppm of hydrogen.

The cantilever beam stiffness tester is being modified so that angular deflection and bending moment will be recorded rather than determined by visual inspection.

Impact and stiffness specimens exposed in-pile to circulating process water at 120°C and 150°C for about one month have been discharged and will soon be tested.

The rate of growth of Zircaloy-2 sheet over the temperature range 500-700°C in air can be expressed by the equation "per cent growth = $K(\text{time})^a$ " where K is a constant dependent on the temperature and the best estimate for a over the temperature range considered is 1.34. A linear relationship exists between the rate constant K and the reciprocal of the absolute temperature. This fits an Arrhenius type equation $K = A \exp(-Q/RT)$, where Q , the activation energy, was $64,600 \pm 23,000$ calories/mol., and $K = 8.96 \times 10^{-12} \exp(-64,600/RT)$. Extrapolation of these data to 350°C indicate that 300 years are required for 0.10 percent growth.

The weight gain of Zircaloy-2 in air at 600°C was determined by in situ weighing. The rate of weight gain conforms to the equation $W = Kt^a$ over a period of 500 hours, where a equals one. This differs from other work where values of a varied from 0.3 for crystal bar to 1.5 for Bureau of Mines zirconium. Further tests are planned to recheck the value obtained.

In-pile oxidation of zirconium in a carbon dioxide-helium atmosphere at about 600°C is much more severe than is the oxidation of Zircaloy-2 under similar conditions. Preliminary examination indicates that the rate of attack in-pile is substantially less than the rate ex-pile. This will be investigated further.

PT-105-524-SI, "In-pile Measurement of Reaction Between Pile Gas Impurities and Proposed Process Tube and Slug Jacket Materials," is being assembled for the mock-up test. The initial tests will take place in two tubes, one with humidified air, the other with dried air.

A mechanism has been proposed to explain the growth of zirconium when exposed to air at high temperatures. The greater volume of the oxide, and its hypothesized high compressive strength are assumed to cause creep and relaxation of the metal, resulting in the growth. A test that confirmed the mechanism consisted of introducing weak points in one surface of a plate by rough grinding, while the other surface remains smooth. The roughened surface breaks up the oxide film so that the compressive stress is limited to the other surface, resulting in pronounced bowing of the plate.

The cold-worked zirconium irradiated to determine the effect of pile exposure on the mechanical properties of zirconium has been partially discharged. Twelve tensile and twelve hardness and metallography wafers were included in this batch. These specimens have been inspected visually after decanning. The second group is still in F pile, and the third group will soon be charged.

Structure Studies

Two prototype Zircaloy-2 capsules containing NaK and uranium were autoclaved at 170°C and 250 and 500 hours. A dark oxide formed during welding was attacked to some extent during autoclaving, but this attack was not serious. This same oxide did not react with $\text{HNO}_3 \cdot \text{HF}$ solution. A test is planned with a drilled capsule which will be autoclaved to determine the extent and nature of the reaction caused by water entry.

The vacuum cathodic etcher performed satisfactorily, developing structure that has been obscured when chemically etched. It was found that the inclusions are etched more slowly than the matrix and a subdomain structure exists in the beta heat treated uranium which could provide boundaries for fission product concentration.

The Faxfilm replica technique was found to be satisfactory for determining that inclusions are preferentially attacked during chemical etching, and by varying the shadowing angle on the replicas it was possible to quantitatively determine the depth to which the inclusions were removed by this preferential attack.

Vacuum annealed disks of aluminum, uranium, zirconium, and AlSi have been cleaned and polished, and are ready for hot pressing to bond the various metals so that their diffusion characteristics can be determined both in and out-of-pile.

Samples of molybdenum and zirconium have been prepared for insertion into the pile to determine the effects of irradiation on the crystal structures of these materials. Pre-irradiation work has been completed, and a fast flux monitor has been designed which appears to be suitable for this experiment. This flux monitor consists of graphite since it has been observed that the c spacing of the graphite lattice increases linearly with integrated fast flux exposure.

Specimens of uranium charged to determine the crystal structure damage at low temperatures have been discharged. More specimens are scheduled for charging in July.

Additional work has been done on the determination of radiation damage in uranium by X-ray diffraction. A sample irradiated to 150 MWD/AT was compared to a control taken adjacent to the irradiated specimen and subjected to the same heat treatment. A definite decrease in the intensities of the (110) and (200) reflections was observed in the irradiated specimen, but no diffraction line shift or line-broadening was evident. It was concluded that any radiation damage present was not of sufficient magnitude to be detected with the diffraction equipment.

A specimen irradiated to 1000 MWD/AT was compared to a powder metallurgy compact, since no adjacent control sample existed. Definite changes in the intensities of certain reflections occurred; this was accompanied by line broadening, which was greater than could be expected as a result of fabrication, and was attributed to radiation effects. No shift in diffraction peak positions was observed; this suggests the damage is due to large fission fragment atoms dispersed at random throughout the lattice. Further work is being done to aid in the evaluation of the data and to provide a clue as to the cause of the observed phenomena.

PILE METALLURGY

Fuel Element Fabrication

Four mechanically bonded Hanford size fuel elements have been prepared for an in-pile test to evaluate the pile performance of slugs fabricated in this manner and to determine the extent of diffusion which may occur at the uranium-aluminum interface during pile operations. An improved cold closure, which eliminates the necessity for a fusion weld at the cap end of the slug, was used for the preparation of these fuel elements. The pile testing of the four mechanically bonded fuel elements is scheduled to begin late in July. The slugs will be irradiated to 300 and 600 MWD/T and will be carefully examined following the in-pile exposure.

Evaluation studies of experimentally prepared cold closures, which are formed by upsetting a heavy can wall section on aluminum jacketed slugs, have indicated that the quality of the point pressure weld is improved by: 1) providing a rough surface on the end of the slug to prevent flow of the aluminum and subsequent flow of oxides from the base of the cap into the point closure; and 2) machining a small recess at the center of the slug end to permit a flushing of part of the oxides out of the plane of the cap. Closures which have been prepared in this manner have been examined metallographically and etched with a 30% sodium hydroxide solution and appear free of oxide stringers throughout the major portion of the cap thickness.

The post-irradiation examination of Hanford fuel elements has indicated that the split-type ruptures which have occurred at HAP0 may be caused by the propagation of a crack or cracks in the uranium core. The propagation of this crack to the uranium surface introduces stresses in the aluminum jacket which are accommodated by a localized strain occurring within a very short gage length and ultimately produces a tensile failure of the jacket material. This situation might be improved by omitting the Al-Si bond between the uranium and aluminum. The gage length of aluminum which would be stressed in event of a can failure in an unbonded fuel element would be increased--perhaps to include the entire circumference of the can--and the probability of jacket failures resulting from the propagation of cracks in the uranium would be reduced. To test this hypothesis, eight unbonded aluminum-jacketed fuel elements are being fabricated for pile testing at HAP0.

Initial attempts to fabricate an insulated fuel element in which the uranium surface temperature will be substantially above that of the pile coolant have been unsuccessful. Several slugs have been prepared by sizing aluminum jackets having an anodized interior onto uranium cores. Examination of these slugs indicated that the extrusion of the can wall during sizing was greater than had been anticipated, and that fragments of the anodized layer were present in the weld area. The sizing operation also resulted in a severe cracking of the anodized layer along the can wall.

Fuel Element Evaluation

Plastic stress calculations have been made to determine the magnitude and distribution of stresses induced in Hanford fuel elements during pile operating conditions. The results of these analyses indicate that the predicated stress values in a cored fuel element operating at 50 kw/ft are appreciably lower than the stresses developed in a solid cylindrical fuel element operating at the same power. These analyses have also indicated that the predicated stress values are highly dependent upon the shape of the stress-strain curve; thus factors such as radiation effects on the mechanical properties, creep relaxation, etc., must be considered in determining, quantitatively, the stresses and strains occurring in fuel elements during pile operations.

Four externally cooled cored fuel elements fabricated from enriched uranium (1.75% U-235) are being irradiated at the highest flux position available in Hanford piles. The calculated power generation of these fuel elements is approximately 75 kw/ft; and as of June 12 they had accumulated an exposure of approximately 1300 MWD/T. The test will be continued until a failure of one of the cored slugs or one of the normal solid slugs in the tube necessitates a discharge of the fuel elements from the reactor.

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The fuel element testing facility, which will permit the irradiation of three four-inch Hanford fuel elements in the MTR at power levels up to approximately 100 kw/ft, has been shipped to the Materials Test Reactor site, and its out-of-pile equipment associated with this facility will be installed in the MTR during the July 28th shutdown and a program of irradiation of Hanford fuel elements in this facility will be initiated. The facility consists of a special aluminum A-block, which contains the fuel elements to be tested, and the necessary external equipment for supplying cooling water and the safe handling of effluent water in event of failure of the test specimens. The fuel elements to be irradiated in the first experiment with this facility include: one cored uranium slug, one standard production Hanford slug, and one fuel element which was assembled by a hot-press canning technique. The second irradiation will consist of three Al-Si canned fuel elements--one a regular production slug and two which differ only in that longitudinal notches have been machined in the surface of the uranium cores to simulate the stress concentration effect of metal defects.

Fuel and Producer Materials

Specimens of an uranium-magnesium fuel material are being prepared for high burn-up exposures in the MTR. The twelve samples will be canned in zirconium and irradiated to burn-ups corresponding to 1000, 5000, and 10,000 MWD/T. The post-irradiation examination of these specimens will include metallographic examination and bend testing to determine the effect of irradiation on the properties of this fuel material.

A study of the solubility of molten magnesium in zirconium has been initiated to determine the feasibility of casting a uranium magnesium fuel material directly into zirconium cans. The examination of zirconium samples which were exposed to molten magnesium at 750-800°C indicates some limited solubility of magnesium in zirconium exists at these temperatures.

A shot tower has been fabricated and installed to determine the feasibility of producing spherically-shaped uranium metal particles for use in the proposed U-Mg fuel material.

A combined fuel-target element for the production of U-233 has been proposed which consists of a thorium core surrounded by a thin foil of oralloy or an oralloy-aluminum sheath. A small number of test pieces will be fabricated to determine the nuclear properties of this fuel element. An approximate calculation has been made of the strain developed in the jacket of such a slug if the gaseous fission products escaped from the oralloy layer after a 30 percent burn-up. A radial displacement of approximately 27 mils is predicted in the case of an unbonded 35 mil aluminum jacket.

Three uranium metal specimens which were irradiated for 24 hours in a HAP0 reactor at calculated temperatures of 660, 800, and 900°C have been visually examined. No evidence of bumping, warp, or growth of the specimens was apparent. A more detailed metallographic examination of these specimens is in progress.

Bids have been received for the off-site construction of a pressurized water loop. This facility will be used for a study of the reaction between 300°C water and uranium metal heated to 700°C.

The time-temperature relationship for the transformation of a gamma-quenched uranium - 14 atomic per cent molybdenum alloy has been determined. Gamma-quenched specimens of this alloy have been annealed at 150, 275, 350 and 400°C. The samples were removed from the furnace at regular intervals and examined to determine whether or not transformation had occurred. A significant change in the X-ray diffraction patterns and hardness of the alloy occurs during transformation. The times required to produce these changes for samples annealed at 275, 350, and 400°C were 228, 112, and 24 hours respectively.

Recycle of Depleted Hanford Uranium

A study has been initiated to determine possible methods and the feasibility of recycling depleted Hanford uranium for plutonium production. The experimental program includes a study of: (1) processes for the reduction of uranium halides and oxides to metals, and (2) the preparation and evaluation of oxide ceramics as fuel material. Several reductions of UO_3 to uranium metal have been made using aluminum and calcium as reductants. In addition, sintered compacts of UO_3 and UO_2 having densities of 4 gm/cm and 6.4 gm/cm respectively have been prepared for reactivity studies.

RADIOMETALLURGY

Examination

Radiometallurgical examinations proceeded normally throughout this period. Metallographic and hardness studies of a transverse section from an irradiated, enriched slug (1.75 percent U-235) provided confirmation of previous calculations that the center of the slug would be heated above the alpha beta phase during a special irradiation test. Another similarly enriched slug developed a meandering indent in the side wall of the can during its irradiation. A transverse section through the center of the slug revealed a fine crack in the uranium beneath the indent and also that the AlSi bending layer had been cracked.

Ruptured slugs from two production tests were examined to determine the cause of failure. In two cases, a uranium defect was noted in the form of a longitudinal crack which probably existed prior to the canning operation. Four ruptures from salt bath beta heat treated, Fernald rolled rods were carefully examined since they represented a resurgence of the cap type of failure. Observations revealed that the three available cap assemblies were solid, the four uranium slugs were concave at one or both ends, the four uranium slugs were eccentrically canned, the overall length of all the uranium slugs had increased, and core rupture had a carrot-shaped cavity extending ~ one inch from the end of the slug without breaking the metal into a compound cap type of failure.

Separation of preformed metallurgical production test samples from their containers to determine the effect of irradiation on uranium is continuing. A few hardness surveys duplicated previous findings of a slight increase resulting from irradiation. In anticipation of a continuation of this test, pre-irradiation measurements of density were made.

Macroscopic and microscopic examination of KEL-F plastic samples after exposure to Purex test solutions revealed an increase in surface pitting and internal flaws in proportion to their exposure.

Facilities

The temporary facilities at the 111-B Building were returned to the Manufacturing Department after removing as much of the deposited radioactivity as was possible.

The general cell facilities were improved by providing a better waste disposal system for filtered liquids and by dividing the slug examination cell into two equal shielded compartments. The cut-off facility demonstrated the ability to successfully obtain longitudinal sections from irradiated uranium slugs up to 2 1/2-inches long. A vacuum furnace was installed in an intermediate level cell and design of another vacuum furnace for tensile testing completed. A bend test jig was designed to obtain strength and ductility of samples cut from standard irradiated slugs.

Metallographic equipment was improved and fabrication of equipment to do cathodic etching and chemical macroetching completed.

Hardness testing was successfully accomplished with the installation of a Wilson Remote Operating Hardness Tester and positioning grips.

In the process of design or modification is equipment to provide small punchings from irradiated samples for chemical analyses; to determine the fission product cross section of a Hanford slug; to provide measurements of the thermal conductivity, electrical resistivity, and density.

WELDING AND CORROSION

Comparison of the Corrosive Effect of Cr^{VI} in Nitric Acid and in 70% Aluminum Nitrate

A review of the data on the effects of Cr^{VI} in nitric acid solutions revealed that when the corrosion rates determined in the first 48 hour boiling periods were plotted against Cr^{VI} concentration in each nitric acid solution, a family of straight lines resulted. These lines were very nearly parallel and were displaced toward higher corrosion rates as the nitric acid concentration was increased from 10 to 60% by weight. A similar line was determined for the effect of Cr^{VI} in 70% aluminum nitrate and its position and slope indicated that (1) the corrosivity of Cr^{VI} bearing 70% aluminum nitrate is less than that of Cr^{VI} bearing 10% nitric acid, and (2) that the effect of increasing the Cr^{VI} concentration is less serious in 70% aluminum nitrate than in nitric acid solution of concentrations 10 to 60%.

Redox Waste Storage Tank Corrosion Test

Some results of a field corrosion study of SAE 1020 carbon steel exposed to Redox process waste solution have recently become available. From the test it was learned that the extent of pitting attack after extended exposure (9 months) is not serious. The deepest pit observed was approximately 0.003 inches deep. The uniform corrosion rates for steel exposed to the waste show a maximum of 0.00001 inches penetration per month. No cracks were observed at 2X and at 200X magnification on the single corrosion test specimen which was stressed to near the yield point.

Redox Waste Concentration Corrosion Studies

Laboratory corrosion tests of types 304L and Carpenter 20 stainless steel and of commercially pure titanium have been continued in both the present Phase I and in the proposed Phase II Redox D-12 Waste Concentrator bottoms.

The tests show that the Phase II solution used in this series of tests is not as corrosive to type 304L stainless steel as Phase I solution when exposures are carried beyond the 5th period; however, the corrosion rate is excessive in either case.

Titanium and titanium welds have shown excellent corrosion resistance to the Phase II solution, as well as to the Phase I solution.

Pilot Plant Heat Exchanger Studies

The second series of heat exchangers are currently being tested in the F4 vessel, 321 Building. These tests are designed to evaluate the following factors:

1. Relative corrosion resistance of new fabrication materials.
2. Desirability of various fabricating techniques.
3. Design variables.

New materials of interest being tested in this series of Heat Exchangers are types 312 and 329 stainless steel and titanium. The tests started on June 14, 1954, and should be completed about September 15, 1954, a three month period.

Bearing Studies

Specimens of Tungsten Carbide, Tantung, V2B alloy and carbolube have been exposed to boiling 65% nitric acid as a preliminary screening test to determine their suitability for use as process solution lubricated bearing surfaces. Of the four materials tested only the carbolube AP-80 plain resisted the corrosive attack of the test media sufficiently to warrant possible application.

Examination of Tube Bundles for the Redox Waste Concentrator (D-12) and Oxidizer (H-4)

Seven bayonet type tube bundles which have been fabricated for service in the Redox Waste Concentrator (D-12) and/or Oxidizer (H-4) have been examined and found to contain defects which may shorten their service life.

Weldability Tests of High-Strength Low-Alloy Steels

The Metallurgy Unit was requested by Reactor Design and Development Unit to perform weldability tests on three high-strength low-alloy steels which are being considered for materials of construction in replacement downcomers at B and D reactors, program CG-558.

The tests indicate that alloys Mayari-R and Yoloy when welded produce ductile joints and the materials are not notch sensitive. Mayari-R appears to be better than Yoloy, and Cor-Ten appears to be unacceptable as the base material fails in a brittle cleavage type fracture.

PHYSICAL METALLURGY -- S. H. BushThe Effects of Irradiation on the Properties of Uranium - R. E. Hueschen, R. S. Kemper, L. J. Chockie

The objective of this program is to evaluate quantitatively the effects of neutron flux intensity, integrated neutron flux, irradiation temperature, testing temperature, and post-irradiation annealing on the properties of uranium.

Four preformed tensile specimens which were irradiated to an average exposure of 310 MWD/AT (PT 105-3N) were tested in the Radiometallurgy Laboratory. Two of the specimens were tested in the as-irradiated condition and two were vacuum annealed at 400°C for 15 hours prior to testing.

The specimens used for this study were obtained from a normal uranium rod which was rolled at the Simonds Saw and Steel Company in November 1952. The rod was machined into eight-inch sections and each section was given the standard beta heat treatment by the metal bath triple-dip process. The unirradiated control samples and the specimens irradiated for this study were machined in pairs from adjacent sections of this one rod. The chemical composition of representative sections along the length of this rod is given in Table I.

TABLE ICHEMICAL COMPOSITION OF URANIUM METAL USED FOR THIS IRRADIATION STUDY

<u>Element</u>	<u>Composition - ppm</u>	<u>Element</u>	<u>Composition - ppm</u>
Carbon	654 - 686	Iron	36 - 45
Nitrogen	40 - 55	Silicon	26 - 50
Hydrogen	1.1 - 1.8	Manganese	10
Aluminum	50	Magnesium	10 - 20
Chromium	10 - 20		

The exposure of 310 megawatt days per adjacent ton obtained for the uranium specimens included in PT 105-3N was obtained from the average heat generation of the surrounding uranium tubes and an assumed cosine flux distribution along the tube.⁽¹⁾ This type of exposure determination does not take into account the decrease in flux depression which exists because of the small (approximately 0.5-inch diameter) uranium samples which were irradiated. The Physics Unit has completed an investigation of this "rise factor" related to the exposure of these preformed specimens using their exponential pile.⁽²⁾ It was concluded from this experiment that the average number of fissions per unit volume is twice as great for 1/2-inch diameter uranium specimens as it is for adjacent standard slugs of uranium in an 8 3/8-inch wet lattice. The application of this factor to the adjacent ton determination of 310 MWD/AT gives a corrected exposure for these smaller diameter specimens of approximately 620 MWD/T.

(1) Lloyd, R. C., "Rise in a One-Half Inch Uranium Rod Loaded into a B,D,F, Type Lattice," HW-32333, July 6, 1954, Secret.

(2) Clark, W. H., "Exposure of Samples of Special Irradiation Request PT 105-3N," HW-30692, January 28, 1954, Secret.

The stress-strain curves obtained for the unirradiated and irradiated tensile specimens are shown in Figure 1. The initial portions of the curves are straight lines up to approximately 22,000 psi for the as-irradiated, and 18,000 psi for the irradiated annealed specimens. The modulus of elasticity values obtained were $30\text{--}33 \times 10^6$ psi for the as-irradiated, and $28\text{--}30 \times 10^6$ psi for the irradiated-annealed specimens. The yield strength, using the 0.1 percent offset method, increased by more than a factor of two after an exposure of approximately 620 MWD/T.

The vacuum annealing of two of the irradiated specimens at 400°C for 15 hours did not cause a significant recovery of the damage; this is shown by the results of specimens 4A and 5A as given in both Figures 1 and 2, which indicates that work-hardening type damage is insignificant compared to the "foreign atom" effect caused by fission product concentration and distribution. This "foreign atom" effect could be analogous, in some respects, to precipitation or age-hardening of alloys. In addition, these data also indicate that the effect of hydrogen is also small in comparison to the total damage, since hydrogen would be released at the pressures and temperatures of the annealing treatment.

The unirradiated tensile data are presented graphically in Figure 2, together with the irradiated stress-strain curves, to illustrate the marked decrease in ductility and toughness caused by the irradiation. Compared to the unirradiated control specimens the ductility, as measured by percent elongation, decreased by a factor of 50 for the as-irradiated specimens, and by a factor of 35 for the irradiated annealed specimens. The energy of rupture, given by the area under the curves of Figure 2, indicates the marked decrease in toughness due to the irradiation. The average values calculated for the energy of rupture are 155, 205, and 12,500 inch pounds per cubic inch for the as-irradiated, irradiated annealed, and the unirradiated control specimens, respectively.

The mechanical property data obtained in this study are given in Table II. The elongation values for the irradiated specimens include both elastic and plastic deformation since they were measured entirely with the extensometer to the point of fracture. The actual bench elongation would be somewhat less than the percent elongation given in Table II. Three of the four irradiated specimens broke near the center of the gage length. The last specimen tested in this study, 4A, broke at the taper gage length juncture, and the value given for percent elongation is possibly somewhat low. It is interesting to note that the ultimate strength of the irradiated samples is lower than for the unirradiated control specimens. All of the four irradiated tensile specimens exhibited the typical brittle fractures of a hard material.

Supplement A of PT 105-3N is a continuation of the study of the effects of irradiation on the physical and mechanical properties of uranium. Supplement A involves the irradiation of a similar amount of test samples to three other exposures in a Hanford pile, exposures to approximately 75, 150, and 1200 MWD/AT, to augment the original 300 MWD/AT exposure of PT 105-3N. Sufficient samples have been fabricated to allow three values to be averaged so that a statistically reasonable curve can then represent the data.

The possibility of using liquid sodium-potassium alloy as the heat transfer medium during the irradiation of the specimens was investigated, with the decision reached that NaK canning of the assemblies for Hanford irradiation was not entirely feasible at this time. The canning method that is being used to

FIGURE 1

FIGURE 2

COMPARISON OF MECHANICAL PROPERTIES OF UNIRRADIATED & IRRADIATED URANIUM METAL			
SPECIMEN HISTORY (ALL SPECIMENS FROM SAME URANIUM ROD)	ULTIMATE TENSILE STRENGTH-PSI	YIELD STRENGTH PSI	ELONGATION % IN 1 INCH
UNIRRADIATED CONTROL SPECIMENS	104,000	31,000(0.1%) 35,000(0.2%)	17
SAMPLE 2-A AS IRRADIATED TO AVERAGE EXPOSURE OF 310 MWD/AT	74,500	67,000(0.1%)	0.36
SAMPLE 4-B AS IRRADIATED TO AVERAGE EXPOSURE OF 310 MWD/AT	77,500	76,000(0.1%)	0.36
SAMPLE 4-A IRRADIATED & VACUUM ANNEALED AT 400°C FOR 15 HOURS	65,500	55,000(0.1%)	0.44
SAMPLE 5-A IRRADIATED & VACUUM ANNEALED AT 400°C FOR 15 HOURS	65,000	49,000(0.1%) 61,000(0.2%)	0.54

TABLE II

assemble the specimens for irradiation is nearly the same as was used for the original exposure of PT 105-3N, the method described in detail in HW-30114.⁽³⁾

The canning method consists of surrounding the preformed sample with tight fitting aluminum inserts and sealing the assembly in a standard HAP0 can. The gap between the uranium sample and aluminum insert is a nominal six mils, which is filled with helium as the heat conducting medium. The assemblies for Supplement A are modified so that all surfaces of the inserts that contact the uranium are coated with colloidal graphite to prevent the diffusion bonding of the uranium to aluminum that was experienced with the first exposure of PT 105-3N,⁽⁴⁾ and all edges are rounded to facilitate the post-irradiation disassembly. Another modification of the canning technique is the use of two 0.1-inch caps on the sized assemblies; in the event that the first fusion-weld closure is rejected by radiography the first cap is removed in a lathe and a second welding attempt made on the second cap. The use of the two caps has reduced the necessity of re-assembly after a reject weld by nearly fifty percent. Complete weld inspection is by radiography, bubble testing, and high temperature, high pressure autoclaving.

The Effects of Low Temperature Irradiation on the Properties of Uranium - R. E. Hueschen, R. S. Kemper, L. J. Chockie

Work has continued on the program to determine the effects of irradiation at 10^{16} to 3×10^{18} nvt with maximum specimen temperatures of approximately 50°C.

Thirty-six tensile and thirty-six electrical resistivity specimens have been canned by encasing them in flattened aluminum tubes sealed at both ends. These cans have been autoclaved to check the closure and then prepared for pile exposure by assembling in slotted 2S aluminum dummy slugs. A typical specimen can and assembly is shown in Figure 3.

These specimens will be irradiated as soon as the selection and scheduling of pile facilities is completed.

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

The initial irradiations at the MTR have been planned for preformed tensile and impact specimens with the test regions of the samples at approximately 400°C during irradiation. The samples will be canned in Zircaloy-2 capsules with NaK as the heat transfer medium. Four 1/4-inch diameter, 1-inch gage length tensile samples and four Izod impact specimens will be canned for irradiation of two samples of each type at exposures of approximately 600 MWD/T and 1500 MWD/T.

The test specimens and capsules have been prepared and tests of the material before irradiation are being conducted. Heat transfer calculations for the assemblies at several conditions have been completed by the Heat Transfer Sub-Unit. The results of these calculations indicate that slight changes in the specimens will be required to obtain the desired temperature conditions during irradiation.

(3) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," HW-30114, January 25, 1954, Secret.

(4) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

Photograph Unclassified

FIGURE 3

SPECIMEN CAN AND CONTAINER ASSEMBLY ILLUSTRATING METHOD OF EXPOSURE FOR FLAT URANIUM SAMPLES.

Zirconium Embrittlement - S. H. Bush, L. F. Kendall, R. G. Wheeler

The embrittlement of Zircaloy-2 by hydrogen is being investigated, using impact and stiffness testing methods. Impact specimens were homogenized at 1000°C for one hour, then furnace cooled. During the furnace cooling one specimen had approximately 200 ppm of hydrogen added. Another specimen was furnace-cooled with no hydrogen added. These and two as-received specimens were impact tested in a 240 ft-lb Charpy machine with the following results:

<u>Conditions</u>	<u>Test Temperature °C</u>	<u>Impact Strength ft-lb</u>
As-received	25	13
As-received	190	26
Furnace-cooled	25	9.5
Furnace-cooled-200 ppm H ₂	25	4

Fractures differed markedly due to grain coarsening and change in mode of fracture. The coarse grained furnace-cooled specimens were examined metallographically at 250X. The fracture in the sample containing hydrogen was predominantly intergranular, while that of the coarse grained specimen without added hydrogen was predominantly transgranular. Micrographs of these specimens are shown in Figure 4.

A cantilever beam type stiffness tester is being used in conjunction with impact tests to evaluate hydrogen embrittlement of zirconium. The stiffness tester is being modified from a visually read instrument to one that will record both the angular deflection and bending moment. This modification should facilitate testing of the irradiated specimens, as well as minimize errors in the data from which brittle to ductile fracture transition temperatures are determined. Brittleness criteria such as angle of deflection at fracture and area under the deflection moment curve were found to be sensitive to human errors in reading the stiffness tester visually. Tests are being conducted to evaluate the statistical significance of embrittlement data obtained with the stiffness tester.

A number of impact and stiffness test specimens have been exposed in-pile to circulating process water at 120 and 150°C for about one month. These specimens are now available for embrittlement testing.

Ex-pile Reaction of Zirconium and/or Zircaloy-2 with Gases - L. F. Kendall

Observation of the increase in length of specimens of Zircaloy-2 sheet heated in air at various temperatures has continued. (Experimental details are described in Reference (5)). The observed data have been found to fit an exponential expression:

$$\% \text{ growth} = k(\text{time})^a$$

where k and a are constants for a given temperature. Values of the constants for various temperatures as determined by least squares analyses are listed in Table III.

- (5) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, p. 50, Secret.

(a) INTERGRANULAR FRACTURE - 250 ppm H₂ ADDED.

Photographs Unclassified

(b) TRANSGRANULAR FRACTURE. NO H₂ ADDED.

FIGURE 4

SECTION OF ZIRCALOY-2 IMPACT SPECIMENS SHOWING
FRACTURE. POLARIZED LIGHT. MAGNIFICATION 190X

TABLE III
VALUES OF CONSTANTS FOR EXPONENTIAL EQUATION
% growth = Kt^a

<u>Temperature °C</u>	<u>k, % per hour</u>	<u>a</u>
500	3.2×10^{-5}	1.58
550	2.0×10^{-3}	1.23
575	3.2×10^{-3}	1.24
600	1.1×10^{-3}	1.56
635	4.4×10^{-2}	1.26
675	1.5×10^{-1}	1.22
700	11.9×10^{-1}	1.28

A statistical examination of the data indicates that the values for a are not significantly different and that the best estimate of a is 1.34 ± 0.39 (95 percent confidence limits).

After plotting the logarithm of the rate constants versus the reciprocals of the absolute temperatures, the equation of the best straight line through the points was calculated by the method of least squares. Using the Arrhenius-type equation, $k = A \exp(-Q/RT)$, the activation energy was calculated to be $64,600 \pm 23,000$ cal/mole (95 percent confidence limits). The rate constant in (per cent)^{-1.34} per hour is $k = 8.96 \times 10^{12} \exp(-64,600/RT)$.

Extrapolation of these data to lower temperatures shows that at 350°C, for example, about 300 years are required to accumulate one-tenth percent growth. At lower temperatures, of course, still larger times are required.

A single experiment was run to determine the weight gain of Zircaloy-2 in air at 600°C. A specimen of 1/16-inch sheet was hung inside a vertical tube furnace by means of a wire fastened to one end of a balance beam. The weight gain per unit original area was found to be a linear function of the time up to 500 hours; i.e., a is one in the expression

$$w = kt^a.$$

Phalnikar and Baldwin⁽⁶⁾ using Bureau of Mines zirconium found that at the same temperature a was about 1.5 between 20 and 70 hours. These values contrast markedly with those of Belle and Mallett⁽⁷⁾ who found a equal to 0.3 for the oxidation of crystal bar zirconium between 575 and 950°C and those of Mallett, Belle, and Cleland⁽⁸⁾ who found $a = 0.5$ for the reaction of pure nitrogen at 975 to 1640°C.

Further tests on these problems await completion of better facilities for temperature and gas control.

- (6) Phalnikar, C. A., Baldwin, W. M., Translation ASTM, Vol. 51, p.1038, (1951).
- (7) Belle, J., Mallett, M. W., J. Electrochemical Society, Vol. 101, p. 339, (1954).
- (8) Mallett, M. W., Belle, J., Cleland, B. B., J. Electrochemical Society, Vol.101, p. 1, (1954).

In-Pile Reaction of Zirconium and/or Zircaloy-2 with Gases - R. G. Wheeler

A zirconium wire that was exposed in a dry channel in the C pile at about 600°C was compared visually with Zircaloy-2 specimens that had been exposed in the F pile and heated to about the same temperature. The scaling of the zirconium wires in the C pile was greater than the scaling of Zircaloy-2 specimens exposed in the F pile even though the F pile gas was nominally 90 percent CO₂, 10 percent He, and C pile was nominally 70 percent CO₂, and 30 percent He. It appears from visual observation of these specimens (to be supported later with radiometallurgical examination) that pile irradiation retards the reaction between zirconium or Zircaloy-2 and a mixture of CO₂ and He.

Production Test No. 105-524-SI, "In-pile Measurement of Reaction Between Pile Gas Impurities and Proposed Process Tube and Slug Jacket Materials," has been written and the equipment is being assembled for a mock-up test. The first in-pile test will be run with a dry air atmosphere in one tube and humidified air in the other. This will represent a severe condition of exposure and will provide data which can be compared with available ex-pile data. Subsequent tests will be designed to isolate the effect of various gases or combination of gases and to simulate actual service conditions.

The Effect of Surface Condition on Zirconium Growth - R. G. Wheeler

The phenomenal growth exhibited by zirconium when it is oxidized in air has been mentioned by a number of authors^(9,10,11,12) and studied in detail by Phalnikar and Baldwin.⁽⁹⁾ Kendall⁽¹⁰⁾ has measured the growth rates of zirconium and Zircaloy-2. Since growth of zirconium during oxidation takes place without a corresponding change in density it must be explained by some mechanism other than anisotropic volume change of the metal.

Recent tests wherein plates of zirconium have bowed in a predictable direction as a result of differences in surface geometry indicate that the product of reaction between zirconium and air tends to occupy a larger area than the original metal surface and thus causes the zirconium to yield plastically.

Hayes and Roberson⁽¹¹⁾ have indicated that the breakdown of zirconium held in air at 900°C is a surface phenomenon. Evidence that certain oxide films have a neutral area after removal from the metal surface on which they were formed, that is, different than the area of the oxide when it is on the metal has been pointed out by Evans.⁽¹³⁾

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- (9) Phalnikar, C. A., Baldwin, W. M., Jr., "The Scaling of Zirconium in Air," ASTM, Vol. 51, p. 1038, (1953).
 - (10) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW 31799, May 10, 1954, Secret.
 - (11) Hayes, E. T., Roberson, A. H., "Some Effects of Heating Zirconium in Air, Oxygen, and Nitrogen," Journal of Electrochemical Society, Vol. 96, p. 142, (1949).
 - (12) Dravnieks, A., "The Kinetics of Zirconium-Nitrogen Reaction at High Temperature," Journal American Chemical Society, Vol. 72, p. 3568, (1950).
 - (13) Evans, U. R., Metallic Corrosion Passivity and Protection, Edward Arnold and Company, London, pp. 18, 74, 79, (1946).

Quantitative measurements of the direction and magnitude of stresses in iron and magnesium oxide have been made by P. D. Dankov and P. V. Churaev.⁽¹⁴⁾ By evaporating iron and magnesium films on mica plates, subsequently oxidizing the metal, and then noting the curvature of the plates, these investigators calculated stresses as high as 5600 psi compression for iron oxide and 4100 psi tension for magnesium oxide. Apparently the film formed by the reaction of zirconium with air is under compression and sufficiently strong to cause relatively large sections of underlying metal to yield plastically. If the stress required to cause zirconium to creep at a rate equal to the observed growth rate and the ratio of cross-section of sound oxide to metal are known, the stress in the oxide necessary to cause growth can be calculated. On a specimen of Zircaloy-2 that grew at an average rate of one percent in 17 hours at 700°C, the ratio of sound oxide to metal was 1:16. The stress required to cause zirconium to yield at this rate was extrapolated from creep data, Figure 5, and found to be between 1000 and 3000 psi. The compressive stress in the oxide would therefore be between 16,000 and 48,000 psi.

An oxide film under compressive stresses of this order of magnitude should fail if weak points were deliberately introduced into the surface, and these failures should cause a change in the zirconium growth rate. Weak points in the oxide film should occur at the apex of marks made with a pointed machine tool or with a coarse grinding wheel. Evidence that oxide on zirconium does crack at the apex of sharp tool marks is shown in Figure 6. No cracks appear at the root of the tool marks because the metal underneath supports the oxide film.

A strip of zirconium containing properly oriented lines of weakness on one surface and a relatively smooth surface on the other side should curve when it is oxidized so that the lines of weakness are on the concave side. This effect was shown with three test plates of Bureau of Mines zirconium (4" x 3/8" x 1/8") cut from the same sheet. One piece was ground on one side so that the grinding marks were perpendicular to the long axis and the other side was lapped smooth. A second piece was prepared the same way except that the coarse and smooth side was reversed with respect to the first specimen. The third piece was lapped smooth on both sides as a control. These pieces were first vacuum annealed at 700°C and then oxidized in air at 680°C. After a few hours of heating in air, the two specimens that were ground on one side were obviously bowed with the ground surface on the concave side. The control piece that had the same type of surface on both sides remained flat throughout the test. Figure 7 shows the radius of curvature of the specimen as a function of weight gain and time. Examination of sections through the oxide revealed a smoothing of the original surface contours as the interface between the oxide and the metal moves inward. This healing of the weak points may account for the tendency for bending to stop after a number of hours even though growth continues.

Effect of Irradiation on the Mechanical Properties of Zirconium - R. S. Kemper

Additional work has been done to determine the effects of irradiation on the mechanical properties of previously cold worked specimens of Bureau of Mines zirconium exposed to pile radiation.

(14) Dankov, D., Churaev, P. V., "The Effect of Deformation of the Surface Layer of a Metal During Oxidation," Doklady Akad Nauk SSSR, Vol. 73, p. 1224, 1950, Technical Translation 245.

Photograph Unclassified

FIGURE 6

APEX OF TOOL MARKS ARE CRACKED.
ROOT OF TOOL MARKS (OUT OF FOCUS)
ARE NOT CRACKED.

FIGURE 7 - RADIUS OF CURVATURE AS A FUNCTION WEIGHT GAIN AND TIME, TEMPERATURE 680°C. SPECIMEN 1 AND 2 COARSE GROUND ON CONCAVE SURFACE, LAPPED ON CONVEX SURFACE.

The first group of samples (twelve tensile specimens and twelve hardness and metallography wafers) were discharged from F pile after an average exposure of 190 MWD/AT. The specimen containers for this group have been opened by Radio-metallurgy Sub-Unit personnel, allowing visual inspection of the samples. The second group of specimens is still being exposed in F pile; scheduling for the charging of the third group will be completed soon.

Electron and Optical Microscopy - T. K. Bierlein, D. R. Green

The development of techniques and the design and testing of related equipment required for the microscopic determination of radiation effects in specific materials is progressing.

Two prototype Zircaloy-2 capsules, each containing liquid NaK and an etched uranium specimen have been autoclaved at 170°C for 250 and 500 hours, respectively. A dark oxide layer, which formed near the welded areas of the capsules during their closure, was impervious to the chemical attack of an $\text{HNO}_3\text{-HF}$ solution, but this area did show evidence of a corrosive type attack during the autoclaving test as illustrated by Figure 8. Sectioning, examination, and evaluation of one of these capsules has been completed and indicates that the general technique used for filling and welding is satisfactory and that the irradiation and subsequent microscopic examination of selected, precharacterized specimens is practicable. To ascertain the extent and nature of the reaction caused by water entry into such a capsule, the second capsule will be drilled, then autoclaved to induce failure. A suitable autoclave bomb for this study has been designed by D. C. Kaulitz. The design of this bomb which is now under construction is illustrated in Figure 9. An evacuable, inert atmosphere glove box which will provide greater utility in preparing NaK filled capsules for irradiation than that afforded by the presently used glass vacuum system, has been designed by D. C. Kaulitz and is currently being fabricated. Test uranium specimens for irradiation are now being prepared for the determination of the effect of pile exposure on the microstructure of uranium.

The vacuum cathodic etching unit which utilizes a specimen mount of zirconium sealed directly to glass has been tested and performs satisfactorily. Several slug wafers have been etched with krypton at a pressure of 75 microns, at 3000 volts, and at a current density of about 3 ma/in². The photographs in Figures 10, 11, and 12 represent the final surface structure of alpha rolled, beta heat treated, and gamma extruded uranium, respectively; the specimens were first polished mechanically and then etched cathodically for one hour. Each of the alpha rolled wafers etched to date has shown a characteristic cross of similarly oriented macro grains; with proper illumination the only beta heat treated wafer examined revealed some indication of such a cross; the sole gamma extruded wafer examined showed none. Faxfilm replicas of these specimens were prepared for examination in the electron microscope. A study of these replicas showed: (1) inclusions in the metal etch at a rate much lower than that of the matrix uranium; (2) the etched, alpha rolled wafer is extensively twinned and characterized by pyramid shaped mounds, the orientation and number of which are dependent upon the parent grain; this characteristic is, therefore, crystallographic, and the pyramidal mounds are tentatively interpreted as lamellar twins, the long direction of which intersects the specimen surface; (3) the beta heat treated uranium is characterized by many grains having a mottled, subdomain structure with a linear width of about 0.5 microns; the domain structure of

a. 1.5X

b. 8X

c. 1.5X

Photographs Unclassified

FIGURE 8

PROTOTYPE ZIRCALOY-2 CAPSULE FOR SPECIAL
IRRADIATIONS; a: BEFORE AUTOCLAVING; b and c:
AFTER 500 HOURS AUTOCLAVING

REACTION VESSEL

Photograph Unclassified

FIGURE 9

Autoclave bomb for Zircaloy, U, and NaK destructive testing.

Photographs Unclassified

FIGURE 10

CATHODICALLY ETCHED ALPHA ROLLED URANIUM
a, b, c, ARE BRIGHT FIELD OPTICAL MICROGRAPHS
e, e, f, ARE SILICON MONOXIDE (FAXFILM) ELECTRON
MICROGRAPHS

Photographs Unclassified

FIGURE 11

CATHODICALLY ETCHED BETA HEAT TREATED
URANIUM a, b, c ARE BRIGHT FIELD OPTICAL
MICROGRAPHS d, e, f ARE SILICON MONOXIDE
(FAXFILM) ELECTRON MICROGRAPHS

Photographs Unclassified

FIGURE 12

CATHODICALLY ETCHED GAMMA EXTRUDED URANIUM a, b, c
ARE BRIGHT FIELD OPTICAL MICROGRAPHS d, e, f ARE SILICON
MONOXIDE (FAXFILM) ELECTRON-MICROGRAPHS

beta heat treated uranium may provide boundaries for localized fission product concentration and account for subsequent changes in the properties of irradiated, beta heat treated uranium; (4) the gamma extruded uranium is very coarse textured and is densely populated with mound shaped impurities which appear to be distributed at prior grain boundary regions; such impurities are very similar in appearance to the hemispherical mounds present in both the alpha rolled and beta heat treated specimens.

During the development of suitable methods of uranium surface preparation, it was deemed desirable to establish the degree to which the impurities in uranium metal are attacked by a routine laboratory method used for characterizing the inclusion content of uranium. A uranium specimen was mechanically polished, electropolished in the customary phosphoric acid medium for one minute, then electroetched for 15 seconds in a bath consisting of 10 grams citric acid, 2.5 ml nitric acid, and 490 ml water, at a current density of 15 ma/cm². Four Faxfilm replicas of the surface were taken, and aluminum was evaporated onto the replicas in such a way that the angles of incidence were 85, 80, 70, and 60 degrees respectively. Figure 13 illustrates the appearance of the replicas which show that all of the inclusions have been attacked by the previous treatment. The photographs demonstrate the utility of this method for characterizing depressions, corrosion pits, and topographic features; the length of the shadows on the photographs are direct trigonometric functions of the depth or height of such features.

Diffusion Studies - T. K. Bierlein, D. R. Green

In order to evaluate the effect of pile environment on the diffusion characteristics of Zr/U, Ni/U, Al/U, and AlSi/U couples, it is first necessary to join the components of the couples so that careful microscopic characterization of the very thin initial penetration band will be possible prior to the pile irradiation.

A number of vacuum annealed metal disks, 0.25-inch thick and 0.5-inch diameter, have been lapped and polished, and are now being cleaned of surface contamination by a vacuum cathodic etching technique. Transfer of these specimens, as well as storage and assembly, is done in either a helium atmosphere or vacuum in order to maintain reproducible surface conditions. Hot pressing of the various metal disks at a pressure of 10T/in² and at a temperature of about 500°C for various times will be investigated to determine if it is a satisfactory method for joining such disks. Temperature control and related factors required for the necessary preliminary studies are currently being tested and evaluated.

Since there is much interest in the diffusional characteristics of mechanically bonded diffusion couples during pile irradiation, a suitable prototype specimen holder and external housing capsule has been designed by D. C. Kaulitz. This unit, illustrated schematically in Figure 14, will provide the necessary heat transfer requirements and will prevent lateral flow of metal which would result from the pressure that is required to hold the metals in intimate contact. Such mechanically bonded diffusion couples will yield information regarding the diffusion characteristics of mechanically bonded slugs.

~~SECRET~~

-37-

HW-32236

85 degrees

80 degrees

70 degrees

60 degrees

Photographs Unclassified

FIGURE 13

ALUMINUM SHADOWED FOXFILM REPLICAS OF
ELECTROLYTICALLY TREATED URANIUM. BRIGHT FIELD- 190X

~~SECRET~~

DIFFUSION COUPLE CAPSULE

Photograph Unclassified

FIGURE 14

Diffusion specimens and capsule enclosure for MTR.

Effects of Irradiation on the Crystal Structures of Zirconium and Molybdenum -
R. P. Hurst, W. V. Cummings

After receiving the molybdenum sheet in June it was possible to complete most of the pre-irradiation work required for these studies during the past quarter. To summarize the progress thus far, all the samples have been prepared, the rabbits have been fabricated, and the heat transfer calculations have been made.

A fast neutron flux monitor has been designed which appears suitable for this experiment. According to rather extensive data obtained by the Graphite Group of Pile Technology, the C spacing of the graphite lattice increases linearly with the integrated fast flux exposure.⁽¹⁵⁾ This method is used to determine fast neutron exposures by irradiating small samples. After exposure, the activity of the graphite is low enough so that the C spacing of the graphite lattice can be measured by ordinary X-ray diffraction techniques.

Studies of Uranium Crystal Structure Damage Using Low Irradiation Temperatures -
W. V. Cummings, R. P. Hurst

Four specimens from the experiment designed to study radiation damage to uranium by using low temperature exposures have been discharged from the pile. At the present time they are in storage in the Radiometallurgy Laboratory and will be analyzed as soon as the work schedule permits. Exposures of other specimens have been delayed because of malfunction of the discharging mechanism of the poison column facility. Modifications and repairs to the apparatus have been completed recently and the charging operations are scheduled to be resumed on July 12.

X-Ray Diffraction Studies of Radiation Damage to Uranium - W. V. Cummings,
R. P. Hurst

Since the permanent installation of the double crystal X-ray spectrometer in the Radiometallurgy Laboratory, a great improvement in the performance of this instrument has been obtained. In view of these results, it has seemed advisable to analyze further the radiation damage to uranium which has been reported previously.⁽¹⁶⁾ This previous work was necessarily preliminary in nature because of the temporary condition of the facilities.

The first specimen chosen was from material that had been used in a controlled experiment designed to determine the correlation between dimensional stability and preferred orientation. A uranium wafer, approximately 1/4-inch in thickness was cut from one of the regular Hanford type fuel elements used in this test. This element had received an exposure of approximately 150 MWD/AT which corresponds to a burn-up of approximately 0.017 percent of the total atoms. The temperature of the specimen during irradiation was estimated to be 350°C at the center and 150°C at the surface. The test piece had been subjected to a pre-irradiation heat treatment to assure the annealing and recrystallization of the alpha rolled parent stock. The control sample used for this investigation

(15) Townsend, J. R., Lund, C. A., "X-Ray Diffraction Peak Shapes From Irradiated Graphites," HW-25019, May 12, 1952, Secret.

(16) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," HW-30114, January 25, 1954, Secret.

consisted of a rod section adjacent to the fuel element which was irradiated. This unirradiated control sample was given a heat treatment identical to that received by the test material. X-ray diffraction patterns were obtained for each of the two wafers. A portion of these patterns are shown in Figure 15. It is noted that a slight difference in diffracted intensities exist for some of the crystallographic planes. The most obvious differences are a decrease in the intensities of the (110) and (200) reflections. The positions of all diffraction peaks were measured in an effort to observe any changes in crystal lattice parameters which might have occurred as a result of the irradiation. In addition, the peak profile for several of the higher intensity planes were obtained by manual counting to determine if any broadening of the diffraction lines had occurred. These results are tabulated in Table IV. A study of these data indicate that no diffraction line-shift or line-broadening occurs. It is concluded, therefore, that any radiation damage present in this sample was not of sufficient magnitude to be detected with the diffraction equipment.

The next logical step in the radiation damage investigation was to study a uranium specimen which had been subjected to a longer exposure. A 1/4-inch section, cut from a fuel element that had received an exposure of 1000 MWD/AT (or a burn-up of 0.114 percent of the total atoms) was chosen for this study. Unfortunately, no control sample was available to permit a comparison of pre- and post-irradiation data. The only information regarding the pre-irradiation history of this specimen is that it received the normal production heat treatment of Hanford fuel elements. This heat treatment and the temperature of the sample during irradiation should have been sufficient to anneal any cold work that might have been introduced during fabrication. On the basis of previous experience it was felt, therefore, that any line broadening observed in the specimen would be principally the result of pile exposure. On this basis the diffraction pattern obtained from the sample was compared to one obtained from a specimen cut from an unirradiated powder metallurgy compact. Portions of the patterns are shown in Figure 16. As in the study of the 150 MWD/AT specimen, an investigation of peak positions and line widths was made. Some difficulty was encountered in obtaining exact measurements of peak positions at the higher angles because of low diffracted intensities and background interference. This was an unfortunate circumstance since any change which might have occurred would be accentuated in this region. As a result of this handicap, no conclusion can be reached regarding unit cell size since the errors involved in the measurement of peak positions could conceivably hide a change in crystal lattice parameters.

The measurement of diffraction line width, however, met with considerably more success. Line broadening was evident at the lower angles where the peak-to-background ratio was high and very good measurements could be made. Table V gives a summary of the data obtained. It will be noted, for example, that the broadening of the (131) reflection is 0.12 degrees. This broadening is in excess of any broadening that might reasonably be expected as a result of fabrication and is attributed to radiation effects. The actual profiles of these lines have been recorded and are to be analyzed in an attempt to determine the type of radiation damage which is present.

FIGURE 15

A comparison of X-ray diffraction patterns obtained from irradiated and unirradiated uranium. The two specimens occupied adjacent positions in the parent rod. Sample N-12 is unirradiated material and sample N-11 has received an exposure of 150 MWD/AT.

hkl	2θ (DEGREES)		$\frac{I_{N-12}}{I_{N-11}}$	$B_{1/2}$	
	N-12	N-11		N-12	N-11
020	30.48° ± .01°	30.48° ± .01°	1.0	.20°	.22°
110	34.98° ± .01°	34.97° ± .01°	1.8	.23°	.22°
021	35.58° ± .01°	35.60° ± .01°	1.0	.24°	.22°
002	—	—	—	—	—
111	39.56° ± .02°	39.60° ± .02°	1.0	—	—
130	57.12° ± .02°	57.12° ± .02°	.7	—	—
131	60.34° ± .02°	60.35° ± .02°	1.1	.31°	.32°
040	63.39° ± .04°	63.39° ± .02°	1.5	—	—
200	65.41° ± .04°	—	—	—	—
041	66.49° ± .04°	66.50° ± .04°	1.0	—	—
221	76.80° ± .04°	76.83° ± .04°	1.1	—	—
150	90.48° ± .06°	90.52° ± .04°	.8	.40°	.42°
241	100.76° ± .04°	100.72° ± .04°	.8	—	—
152	101.78° ± .04°	101.84° ± .04°	.9	—	—
060	104.20° ± .05°	104.23° ± .05°	.8	—	—
061	—	107.06° ± .05°	—	—	—
044	109.27° ± .04°	—	—	—	—
062	—	—	—	—	—
135	132.26° ± .04°	—	—	—	—
260	145.61° ± .05°	145.73° ± .06°	.8	—	—
261	151.36° ± .10°	151.20° ± .10°	1.0	—	—

TABLE IV

A comparison of X-ray diffraction data taken from diffraction patterns of irradiated and unirradiated uranium. Specimen N-11 had received an exposure of 150 MWD/AT while specimen N-12 was unirradiated. Originally the two specimens have occupied adjacent positions in the parent rod. In the columns headed 2θ (Degrees), the diffraction peak positions are given for all measurable planes. In addition, the ratios of the intensities and the line breadths at half height are shown in the other columns.

FIGURE 16

A comparison of X-ray diffraction patterns obtained from irradiated and unirradiated uranium. The top pattern was obtained from unirradiated metal which had been fabricated by powder metallurgy techniques. The bottom pattern was obtained from beta heat treated uranium which had received an exposure of 1000 MWD/AT.

(h k l)	2 θ (DEGREES)		$\frac{I_{x-1}}{I_{std.}}$	$B_{1/2}$	
	X - 1	STD.		X - 1	STD.
020	30.47° $\pm$.03°	30.47° $\pm$.01°	.9	—	—
110	34.95° $\pm$.02°	34.98° $\pm$.02°	1.4	.30°	.22°
021	34.59° $\pm$.02°	35.61° $\pm$.02°	.9	.29°	.23°
002	36.25° $\pm$.03°	36.26° $\pm$.02°	.3	—	.23°
111	39.55° $\pm$.02°	39.59° $\pm$.02°	1.0	.30°	.24°
112	51.24° $\pm$.03°	51.28° $\pm$.02°	.4	—	—
131	60.32° $\pm$.03°	60.38° $\pm$.02°	.9	.40°	.27°
040	63.44° $\pm$.04°	63.50° $\pm$.03°	1.0	—	—
200	65.40° $\pm$.04°	65.48° $\pm$.03°	2.3	—	—
221	76.74° $\pm$.05°	76.82° $\pm$.05°	—	—	—
133	83.72° $\pm$.06°	83.80° $\pm$.05°	—	—	—
150	90.58° $\pm$.10°	—	—	—	—
240	97.80° $\pm$.10°	—	—	—	—
223	—	99.05° $\pm$.06°	—	—	—
152	—	101.65° $\pm$.06°	—	—	—
154	—	141.55° $\pm$.10°	—	—	—
312	122.75° $\pm$.10°	—	—	—	—

TABLE V

A comparison of the X-ray diffraction data as obtained from a specimen which had received an exposure of 1000 MWD/AT (X-1) and one which had not been irradiated (Std.). Diffraction peak positions, the ratios of the peak intensities, and the line breadths at half height are shown in the various columns.

Several questions arise from the analysis of these data that can be answered only by further studies, a change in technique, or an improvement in the performance of the X-ray diffraction apparatus used in these experiments. The change in diffracted intensities as was noted in sample N-11 (150 MWD/AT) becomes more interesting in light of the fact that these changes were noted for planes which contribute to plastic deformation and to dimensional instability in uranium. The fuel element from which this sample was cut grew two percent during irradiation. Further investigation is planned to determine whether the intensity change observed is real or apparent; and, if it is a real effect, whether it can be related to a mechanism of dimensional instability during irradiation.

The inability to observe a shift in diffraction peak positions in sample X-1 (1000 MWD/AT) although a definite broadening of the lines was observed, suggests the possibility of large fission-fragment atoms dispersing themselves at random throughout the crystal lattice. The fact that line broadening could not be observed in the lower exposure sample may be indicative that the concentration of fission products was too low to give a measurable effect. As stated previously, the profiles of the broadened lines are being analyzed to aid in the evaluation of the data and to provide a clue to the cause of the observed phenomena. Minor modifications to the diffraction unit are in progress which are expected to give an increase in intensity and provide greater resolution. These improvements could possibly reveal evidence of crystal structure damage that has not been observed with existing equipment.

FILE METALLURGY -- M. J. Sanderson

Mechanically Bonded Fuel Elements - G. A. Last

The four mechanically bonded fuel elements previously described⁽¹⁷⁾ which were to be used for an in-pile test to investigate the possibilities of diffusion at the uranium-aluminum interface successfully passed all pre-pile tests and evaluations. Some doubt now exists, however, as to the quality of the 2S aluminum from which the initial cups were machined, i.e., detrimental stringer inclusions may exist through the base of the cups which in turn may lead to in-pile corrosion difficulties. In order to eliminate this uncertainty, new fuel elements have been prepared using cups machined from 2S aluminum rod which had been pre-checked to insure the non-existence of such defects in the final can.

In light of improvements in the cold closure technique it appears that the fusion weld over the "point" closure, which was used on the initial fuel elements, may well be eliminated as an unnecessary step in the canning procedure. Consequently, no fusion weld is being employed for the slugs which are to be pile tested.

The initial in-pile test of mechanically bonded fuel elements is scheduled to begin in July at HAP0 and will be conducted to exposures corresponding to 300 and 600 MWD/T.

(17) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

Point Closures for Aluminum Jacketed Fuel Elements - G. A. Last, J. E. Minor

Evaluation studies conducted on experimental cold closures have led to marked improvement in the "point" pressure weld obtained by the technique of forming both cap and closure by upsetting of a heavy can wall section.⁽¹⁸⁾ Two factors have been of prime importance in achieving the closure improvement. First, roughening of the end of the slug either anodically or by machining a spiral groove on the end of the slug is beneficial. In either case, the flow of aluminum across the end of the slug is prevented so that the flow of oxides from the base of the cap into the "point" closure is minimized. Secondly, the use of a 0.125-inch diameter by 0.125-inch deep hole in the center of the slug end allows part of the aluminum oxides present at the time of closure to be flushed downward out of the plane of the base of the cap thus minimizing the amount of oxides that have to be extruded out of the cap in order to achieve a sound pressure weld.

Closures formed on slugs having both the dimple and the roughened surface have been subjected to metallographic examination and appear to be free from continuous oxide stringers throughout the major portion of the cap thickness. A 30 percent sodium hydroxide etch, which preferentially attacks inclusions in aluminum, has been used on several closures. A small pit developed at the point of closure on all pieces indicating the presence of oxide stringers. However, from the appearance of the "pit" as etching progressed, it was concluded that continuous stringers did not exist to any significant depth into the cap. This is in agreement with the metallographic observations.

Figure 17 shows the pit which developed at the "point" of closure after the removal of 0.02-inch of aluminum by etching. One-tenth inch of aluminum had been removed from the 0.25-inch cap prior to etching. Figure 18 is a section through the etch pit shown in Figure 17 showing broken oxide stringers remaining in the closure.

Unbonded Fuel Elements - J. E. Minor

In a large fraction of current in-pile slug failures in Hanford piles, a radial or longitudinal crack propagates in the uranium core. The AlSi bond that exists in the current fuel element fails over the crack, but the aluminum on each side of the failure is still bonded by AlSi to the uranium core. Thus, as the crack widens, the length of aluminum stressed by the crack is very short and failure of the jacket quickly follows.

It is hoped that this situation can be improved by omitting the aluminum-uranium bond, leaving the aluminum free to move with respect to the core. In the event of core failure the length of aluminum stressed will then be increased--perhaps to include the entire circumference of the jacket--thus greatly reducing the probability of jacket failure.

Study of the experience in operating water-cooled non-bonded fuel elements at Chalk River leaves little doubt that such an element could be operated successfully at Hanford. With the exception of the length of the fuel element, Chalk River and Hanford geometries are quite similar. The NRX fuel element consists

(18) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - October, November, December, 1953," HW-30896, March 5, 1954, pp. 12-15, Secret.

FIGURE 17

ETCH PIT DEVELOPED AT THE "POINT" OF CLOSURE IN A CAP FORMED BY UPSETTING OF A HEAVY (0.200 inch) CAN WALL SECTION EXTENDING ABOVE A SLUG. TWENTY MILS OF ALUMINUM REMOVED BY ETCHING IN 30 PERCENT SODIUM HYDROXIDE AT 60° C. 6X

FIGURE 18

CROSS SECTION THROUGH ABOVE PIT SHOWING BROKEN OXIDE STRINGER EXISTING IN THE CLOSURE. 80X

Photographs Unclassified

of a ten-foot uranium rod, 1.360 inches in diameter, onto which a finned 0.079-inch thick aluminum tube is drawn. The annulus between the metal sheath and the process tube is 0.100 inches.⁽¹⁹⁾ A water flow of twenty feet per second is maintained during operation.⁽²⁰⁾ The NRX has operated at 30 MW, and it can be calculated that a specific power of 46 kw/ft is achieved at this power level.⁽²¹⁾ Thus, it appears that fuel diameter, jacket thickness, and water flow conditions at NRX are not drastically different from Hanford experience, and that maximum specific powers are comparable in the two cases. These rods have been successfully irradiated to levels as high as 3000 MWD/T. Reports on the few failures that have occurred are incomplete (as of December 1, 1951, six of 583 rods irradiated had failed), but Lavigne has indicated that NRX ruptures are believed to have been due to the inability to detect defective fuel elements before charging rather than to any shortcoming of the jacketing scheme.⁽²²⁾

A "dry" canning method such as that required to produce the non-bonded fuel element has the advantage of easily producing a known can wall thickness. The principal difficulty in canning is the closure. A sound fusion weld between cap and can wall would be difficult to produce without introducing additional material, such as a sealer disk, to prevent access of internal gases to the molten metal during fusion welding. This difficulty has been overcome by the use of the point closure method developed by George Last. This closure can be produced economically and rapidly, and is not affected by access of internal gases to the closure area during the cold welding operation.

Eight unbonded slugs are being fabricated for pile exposure. Two will be irradiated to 200 MWD/T exposure, two to 400 MWD/T exposure, and four to higher exposure.

Insulated Fuel Elements - J. E. Minor, O. K. Shupe

Two of the factors that contribute to the failure of Hanford fuel elements during irradiation are the low ductility of the outer shell of cold uranium and the embrittlement that radiation damage causes throughout the slug. These considerations have led to the design of a fuel element in which the uranium surface temperature has been raised substantially above that of the pile coolant (200-500°C surface temperature). The increased surface temperature will increase the ductility of the outer shell of uranium, and will raise the central uranium temperature sufficiently so that a significant portion of the radiation damage may be annealed out in the slug center.

Of the possible methods of producing a controlled temperature increase between the slug jacket and the uranium, an anodized layer on the inside of the aluminum can has been chosen for initial studies. Properties of such layers are known and application of the anodizing technique to a slug jacket requires relatively little development work. It has been found possible to produce a uniform layer

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- (19) Brooks, H., Howe, J. P., Snyder, T. M., Kingdon, K. H., "Report of Visit to Chalk River, November 3 and 4, 1949," KAPL-281, December 8, 1949, Secret.
 - (20) Lavigne, M. J., "Minutes of the Metallurgy Meeting Held At Chalk River Plant, Building 401, Conference Room, July 13-14, 1948," EM-1, July 20, 1948, Secret.
 - (21) Ozeroff, W. J., "Private Communication to G. A. Last," August 6, 1953.
 - (22) Lavigne, M. J., "Private Communication with M. J. Sanderson at Chalk River Meeting, June, 1953."

of Martin Hard Coat five mils thick on the inside of an aluminum can by anodizing for 80 minutes at a current density of 42 amps/ft² using 15 percent H₂SO₄ as the electrolyte. The electrolyte was cooled with dry ice to maintain a temperature of about 32°F and to maintain a concentration of CO₂ in the bath. In order to maintain a constant current density during the process, it was necessary to vary the applied voltage from 20 to 120 volts. Figure 19 shows the uniformity of the coat obtained on can wall and can bottom.

Two cored slugs have been canned in anodized cans closed by anodized caps. A hot press closure was utilized. Extrusion of the can wall was greater than had been anticipated, and fragments of the anodized layer were found in the weld area. The extrusion of the can wall also produced severe cracking of the anodized layer, and the pressing operation caused aluminum from the can wall to fill these cracks. In the next trial a closer initial fit will be utilized between the slug and can to reduce movement of the can wall. A shorter period of high pressure will be used during the hot press closure to reduce deformation of the core--in the initial attempts the central void in the uranium was partially closed during pressing.

Other anodized cans are being prepared for "C" closure. In these a sealer disk will be utilized between the uranium core and the cap, and the anodized layer will be applied to the sealer disk rather than the cap. The assembly will be sized and a fusion weld applied to the cap.

Thermal Stresses in Fuel Elements - K. R. Merckx

Plastic stress calculations have been made and two methods of analysis have been developed. One method depends upon numerical integration and the other on a variational procedure.

The results of these analyses have indicated that predicted stress values are highly dependent upon the shape of the stress-strain curve (HW-32321). Thus, factors like radiation effects and creep relaxation should be considered in determining the stresses and strains occurring in fuel element operations.

Present work on thermal stresses considers the time and temperature dependent properties of relaxation and creep. To make such solutions, mechanical equations of state are being developed in a form which can be applied to a mathematical analysis and can be evaluated with a minimum amount of experimental effort.

Numerical results of the previous work have indicated a beneficial effect is produced by coring a fuel element. For example, in solid and cored fuel elements operating at 50 kw/ft with a uranium surface temperature of 100°C (HW-31681), the predicted maximum circumferential stresses are:

<u>Ratio of Core Radius To Fuel Element Radius</u>	<u>Temperature Drop °C</u>	<u>Maximum Circumferential Stress - psi</u>
0	380	43,800
0.2	330	35,600
0.4	247	32,400
0.6	162	25,100

Photographs Unclassified

FIGURE 19

SECTIONS FROM ALUMINUM CAN ANODIZED ON THE INSIDE.

OXIDE LAYER

BLACK - Al_2O_3

WHITE - ALUMINUM

200X

Thermal gradients may be reduced by internally and externally cooling a fuel element. The reduced gradients will produce further reductions in predicted fuel element stresses (HW-32322). The analysis in HW-32322 is an elastic solution; hence, direct comparison of these results should not be made with those obtained from the plastic stress analyses.

The following reports are now published describing this work:

- (1) Merckx, K. R., "A Plastic Stress Analysis for Solid and Cored Fuel Elements," Part I: Operation at High Coolant Temperatures, HW-31576, March 31, 1954, Secret. Part II: Operation at Existing Coolant Temperatures, HW-31681, May 12, 1954, Secret.
- (2) Merckx, K. R., "A Variational Method for the Thermal Stresses in an Infinite Cylinder," HW-31651, March 24, 1954, Unclassified.
- (3) Merckx, K. R., "A Variational Method for Calculating the Plastic Thermal Stresses In A Finite Cylinder," HW-32032, Unclassified.

The rough drafts are completed for four additional documents:

- (4) Merckx, K. R., "Plastic Stress Analysis for a Fuel Element with a Core in the Beta Phase," HW-32169, Secret.
- (5) Merckx, K. R., "Elastic Stress Analysis For An Internally and Externally Cooled Fuel Element," HW-32322, Secret.
- (6) Merckx, K. R., "Plastic Stress Analysis For The Thermal Stresses and Strains In Cylindrical Uranium Fuel Elements," HW-32031, Secret.
- (7) Merckx, K. R., "A Variational Method For Determining the Thermal Stresses In An Infinite Plastic Cylinder," HW-32321, Secret.

In-pile Testing of Externally Cooled, Cored Fuel Elements - J. E. Minor

As the power level at which Hanford piles operate has been increased, the number of ruptures attributed to stress failure of the uranium has increased. In this study externally cooled, cored slugs are being tested as a possible replacement for solid slugs in tubes operating at higher powers. An externally cooled, cored slug (in this instance, a slug of standard Hanford outer diameter containing a 3/8-inch axial hole in the uranium), operating at the same specific power as a solid slug, will have a lower maximum uranium temperature and lower stresses in the uranium. Slugs of this geometry should incur a sharply reduced proportion of stress failures.

Four four-inch slugs with 3/8-inch cores were fabricated from uranium enriched to 1.75 percent U-235 and charged in Hanford C pile. The pre-irradiation evaluation of the metal and the method of fabrication and canning were described in a previous report of this series.⁽²³⁾ These slugs, operating at a specific power of about 75 kw/ft, have accumulated an exposure of about 1300 MWD/T and the tube is operating normally. This exposure corresponds to a burnup of approximately 0.15 percent of all atoms in the uranium.

This performance may be compared to that of a columnar charge of solid slugs of the same enrichment operating at only slightly greater specific power. In this

- :
- (23) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - October, November, December, 1953," HW-30896, January 5, 1954, Secret.

test, ⁽²⁴⁾ the highest exposure reached before failure of a slug was 650 MWD/T. By examination of the statistics on exposure levels at which slug failures occur, L. W. Lang ⁽²⁵⁾ has compiled a table correlating the difference in exposure at rupture of test specimens and standard slugs versus the probability that the test specimen represents an improvement over the standard slug. If a full tube test were involved, the probability that cored slugs are less susceptible to split rupture than are the solid would now be 0.99. Only four cored slugs are involved in the test; on the other hand, the slugs are located in the same portion of the tube that the ruptured slugs occupied in the solid slug test.

The cored slug irradiation will be continued until failure of one of the cored slugs or of one of the twenty solid normal slugs in the tube causes discharge.

A cored normal uranium slug has been included in the loading of the first A-block for irradiation in the fuel element testing facility at the MTR. The A-block is scheduled to be loaded in the reactor during the July 7 shutdown.

MTR Fuel Element Testing Facility - T. W. Evans, D. C. Kaulitz, M. C. Fraser

The fuel element testing facility, when completed, will allow the irradiation in the MTR reflector of three four-inch Hanford fuel elements at power levels up to approximately 100 kw/ft, which is a factor of 2-3 times the specific power generation routinely obtained in Hanford reactors. The testing facility consists of a special aluminum A-block ⁽²⁶⁾ and the necessary external equipment for supplying cooling water at pressures up to 350 psi and for safely handling the effluent water if failure of a specimen should occur. A schematic flow diagram of the apparatus is given in Figure 20, and Figure 21 is a photograph of the A-block as it appears before insertion in the MTR.

The design and construction of the testing facility proper has been under the direction of G. E. Wade of Pile Technology Sub-Section. R. S. Paul, also of Pile Technology Sub-Section, has designed and constructed a two-channel gamma ray spectrometer for monitoring the effluent water for fission product activity. At the end of this quarter the testing facility was undergoing a breaking-in period of operation at the MTR with the A-block out of the pile. The A-block is to be charged in the MTR reflector position A-6 on the July 9th shutdown.

The testing facility has been designed to provide several unique features for experiments with the Hanford type of fuel element. Among these are (a) higher specific power levels can be obtained, (b) ruptures can be tolerated, and (c) different geometries can be handled with appropriate changes in the A-block design. As has been mentioned in previous quarterly reports of the Metallurgy Unit, the experimental program will follow the general lines: (1) testing of new fuel element designs, (2) irradiation of defective fuel elements in an

(24) Lang, L. W., "Production Test 105-532-A-4, Irradiation of Enriched Uranium Slugs," January 5, 1954, Secret.

(25) Lang, L. W., "An Examination of In-Pile Fuel Testing," HW-31582, April 28, 1954, Secret.

(26) A sketch showing the internal arrangement appears as Figure 9 in "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," J. J. Cadwell, HW-30114, January 25, 1954, Secret.

**FLOW DIAGRAM
M.T.R. FACILITY**

FIGURE 20

Photograph Unclassified

FIGURE 21

HANFORD A-BLOCK
LEFT - AS IT APPEARS BEFORE IRRADIATION IN THE REACTOR.
RIGHT - DISASSEMBLED TO SHOW THERMOCOUPLE WELLS.

attempt to elucidate rupture mechanisms, especially of the cleavage type, and (3) investigation of the behavior of fuel elements at high specific power levels.

Specimens for the first two irradiations have been prepared, charged in A-blocks and shipped to the MTR. The initial charge contains three four-inch specimens, one solid and one hollow uranium core, AlSi canned, and one solid core canned by the hot-pressing technique, the last specimen being furnished by J. D. Sprowl of Fuel Technology Sub-Section. The purpose of this irradiation is to determine the behavior in the testing facility of Hanford fuel elements of the regular production type and also of two candidates for future use in Hanford reactors. There are several differences between the fuel element environment in the testing facility and that in Hanford reactors, and it is important that the nature of the differences and their effect on the behavior of fuel elements be determined. The principal source of concern at present is the magnitude of the radial flux gradient in the reflector, as has been found, for instance, in the KAPL-15 experiment. Monitor wires placed in the A-block around the specimens will provide information on this point. In addition, the post-irradiation examination will include fission product analysis at several points in the fuel element core if the monitor results indicate that the analysis should be done. It is planned to carry the irradiation to an exposure of 700-900 MWD/T.

The second A-block contains three AlSi canned fuel elements--one regular production piece and two specimens which differ only in that longitudinal notches have been machined in the surface of the uranium cores. It has been long suspected that defects in the surface of the uranium (cracks, seams, striations, etc.) will induce a split type failure of the specimen under abnormal or even normal reactor conditions. The use of the machined notch is an attempt to imitate the stress-raising effect of "natural" defects in a reproducible manner.

Upon completion of the irradiation, the A-block will be opened in the MTR canal, the specimens will be removed, examined visually and then returned to Hanford for metallurgical examination.

Uranium-Magnesium Fuel Material - M. D. Freshley, G. A. Last

Work is continuing on the investigation of a fuel material which consists of discrete uranium particles embedded in a magnesium matrix. As described in previous reports^(27,28) this fuel material would have increased thermal conductivity and dimensional stability and improved resistance to radiation damage.

Three uranium-magnesium fuel elements using uranium in the form of small punchings were fabricated and machined to standard four inch slug size. They were calculated to contain 53.3, 54.05, and 55.0 volume percent uranium. These slugs were canned in aluminum cans with 0.045 inch walls and 0.175 inch end caps. Reactivity tests were conducted on these slugs by M. V. Davis of the Experimental Physics Sub-Unit. The conversion ratio was calculated to be 0.76 corresponding to an infinite K of 1.006. This is equivalent to a reactivity loss of 1000 ± 200

- (27) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - October, November, December, 1953," HW-30896, March 5, 1954, p. 12, Secret.
- (28) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

in hours for a full pile loading in a present Hanford reactor. With enough homogeneous enrichment to restore the reactivity to 1.03, the conversion ratio will drop to 0.73. This is compared to the present fuel element which has a conversion ratio of 0.83 with an infinite K of 1.03⁴. An increase in the volume percent of uranium will improve the conversion ratio and increase the reactivity with less enrichment. Seventy volume percent uranium will be strived for by using small spheres.

A closer examination of the uranium compact impregnated with pure magnesium showed that only about 50 percent of the uranium surface had been wet by the magnesium. The bond strength was not too great as the uranium readily parted from the magnesium. Figure 22 is a photo-micrograph of a pure magnesium-uranium interface.

A compact of uranium turnings was impregnated with an alloy of magnesium - 1.4 weight percent silicon. Examination showed complete wetting of the uranium surfaces with the formation of a compound layer as shown in Figure 23.

Samples of uranium turnings compacts impregnated with pure magnesium and also an alloy of magnesium - 1.4 weight percent silicon were subjected to a boiling water corrosion test and examined microscopically. All samples were boiled for a period of 24 hours. The samples of uranium in a pure magnesium matrix were swelled and badly corroded on the surface, and the water had penetrated into the interior of the specimen as shown in Figure 24. The corrosion products formed in the interior of the specimen were confined by the magnesium. This would explain the gain in weight of these samples.

Microscopic examination of the samples of uranium in a magnesium-silicon matrix showed that the corrosion was limited to the uranium which was exposed to the surface because the corrosion resistant compound layer prevented the penetration of water into the interior of the specimen (see Figures 25 and 26). The corrosion products, in this case, were free to spall off and hence the sample lost weight.

Zirconium was exposed to molten magnesium in the temperature range of 750 to 800°C for different periods of time. This was done to investigate the consequences of pouring magnesium directly into a zirconium can and also the effect of high operating temperatures on the magnesium-zirconium interface. The work was of a preliminary nature and some of the results may have been influenced by the presence of impurities. It can be said, however, that zirconium is soluble in magnesium in the range of 750 to 800°C. Figure 27 shows penetration of magnesium into the zirconium causing intergranular attack. Some work has been done on the magnesium-zirconium phase diagram by various investigators but their work has been of a preliminary nature.⁽²⁹⁾

Samples of uranium-magnesium fuel elements are being fabricated for high burn-up exposures in the MTR facility. The samples will be about one-half inch in diameter and four inches long. Several methods of fabricating uranium chips suitable for these tests were attempted before chips of the proper size were obtained. Their apparent density is about 45 percent of the density of solid uranium. Twelve samples will be canned in zirconium jackets and irradiated. Six will consist of uranium chips surrounded by a pure magnesium matrix and

(29) Smoluchowski, R., et al, "Zirconium Alloy Systems," GEH-21177, Undated, p. 55, Confidential.

FIGURE 22
U - PURE Mg INTERFACE. 190X

FIGURE 23
U - Mg 1.4 W/O Si INTERFACE SHOWING
COMPOUND LAYER. 190X

Photographs Unclassified

FIGURE 24

U - PURE Mg SUBJECTED 24 HOURS
TO BOILING H₂O. 6X

FIGURE 25

U - Mg 1.4 W/O Si SHOWING
CORROSION RESISTANCE OF THE
COMPOUND LAYER. 190X

Photographs Unclassified

FIGURE 26

U - Mg 1.4 W/O Si SUBJECTED 24
HOURS TO BOILING H_2O . 6X

FIGURE 27

Mg - Zr INTERFACE SHOWING
DISSOLUTION OF Zr INTO Mg. 190X

Photographs Unclassified

six will contain a matrix of magnesium - 1.4 weight percent silicon. The tentative plan is to expose the samples for about 1000, 5000, and 10,000 MWD/T. Exposure will be followed by metallographic examination and bend tests to give an indication of the effect of irradiation on the mechanical strength of the material. The use of uranium chips as stand-in material for small spheres is not intended to duplicate the behavior of spheres under radiation, but only to provide an indication of the comparative effects of radiation on the properties of the fuel material.

The graphite spiral shot tower furnace has been assembled and tested and its operation has proven very satisfactory. The furnace is operated in an atmosphere of helium which is purified by passing over uranium chips at 800°C. There was no noticeable oxidation of the graphite element after two successive heatings to above 1350°C. Twenty-five minutes are required to attain this temperature with a power input of about 6 KVA.

Combined Fuel-Target Slug for U-233 Production - G. A. Last, K. R. Merckx

A combined fuel-target fuel element for the production of uranium-233 has been proposed. (30,31) This element consists of a thorium core surrounded by a 2.4 mil or alloy foil or a 42 mil aluminum - 25 weight percent U-235 alloy sheet or tube, which in turn would be canned in an aluminum jacket. A small number of test pieces (using the or alloy foil) are to be fabricated for use in experimentally evaluating the nuclear properties of this fuel element as soon as the or alloy foil can be obtained.

An approximate calculation has been made of the strain developed in the jacket of such a duplex slug if the gaseous fission products should escape from the or alloy layer. A radial displacement of ca. 27 mils is predicted in the case of an unbonded 35 mil aluminum jacket surrounding a 2.4 mil layer of or alloy subjected to an assumed 30 percent burnout.

To simplify calculation, the following assumptions were made:

1. The stress-strain relation is of the form $\sigma = A \epsilon^n$, where the constants A and n can be calculated from the yield strength, tensile strength and modulus of elasticity at 100°C.
2. Strain of the jacket is due exclusively to fission gas pressure.
3. This fission gas pressure results from escape of 100 percent of the stable and long-lived fission product Kr and Xe.
4. The temperature of this gas is only 100°C.

Assumptions 2 and 4 are certainly conservative; nevertheless, the strain calculated appears prohibitive. Accordingly, successful operation of a slug of this design hinges on either (1) demonstrating that fission gas is not released, or (2) establishing an upper limit to permissible burnout well below 30 percent. If the first alternative should prove unfeasible technically and the second, economically, there remains the alternative of enclosing the fuel (enriched U or UO₂) in a matrix (e.g. Al or Mg) in an attempt to confine the fission gas.

(30) Stuart, G. W., "A Fuel Element For Optimum Hanford 23 Production," HW-31727, May 4, 1954, Secret.

(31) Cole, R. K., Stuart, G. W., "Addendum to Report HW-31727 - A Fuel Element For Optimum Hanford 23 Production," HW-32109, June 11, 1954, Secret.

Calculations

burnout = 30 percent
 total yield stable and long-lived Kr and Xe = 24.6 percent
 orally layer thickness = 2.4 mils
 r = slug radius = 0.68 inch
 V_o = volume of gas at STP, cubic inch/inch slug
 p = pressure of gas, psi
 Δr = radial displacement, inch
 t = jacket thickness = 0.035-inch

Amount of fission product gas

$$V_o = \frac{22400 \times 0.25 \times 0.3 \times 2 \times \pi \times 1.36 \times 0.0024 \times (2.54)^3 \times 19}{(2.54)^3 \times 238} = 2.75$$

$$p = p_o \frac{TV_o}{T_o V} = 14.7 \times \frac{373}{273} \times \frac{2.75}{2 \pi r \Delta r} = \frac{8.8}{r \Delta r} \quad (1)$$

Radial Strain

From data in the Metals Handbook, the stress-strain relation is approximated by

$$\sigma = 14600 \epsilon^{0.141}$$

Now $\epsilon = \frac{\Delta r}{r}$ and also $\sigma = \frac{pr}{t}$

Hence $14600 \left(\frac{\Delta r}{r}\right)^{0.141} = \frac{pr}{t} \quad (2)$

Substituting p from eq. (1)

$$14600 \left(\frac{\Delta r}{r}\right)^{0.141} = \frac{8.8}{\Delta r t}$$

$$\Delta r = 2.7 \times 10^{-2} \text{ inch} = 27 \text{ mils}$$

High Temperature Irradiation of Uranium Test Specimens - J. E. Minor, C. L. Boyd

To facilitate study of irradiation effects at high temperatures on the mechanical and physical properties of uranium, an in-pile exposure technique is being investigated which should allow portions of small uranium specimens to attain temperatures in the beta and gamma phase. The type of specimens irradiated and the conditions of irradiation have been described in other reports. (32,33,34)

- (32) Berberet, J. A., "PT 105-508-SI, Irradiation of High Temperature Metallurgical Test Specimens," HW-29268, September 8, 1953, Secret.
- (33) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," HW-30114, January 25, 1954, Secret.
- (34) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - October, November, December, 1954," HW-30896, March 5, 1954, Secret.

Four specimens were charged November 30 and discharged without incident after approximately 26 hours irradiation. The charging and discharging of these specimens was accomplished while the pile was in operation. In this instance, however, the pile shut down for a short time immediately after the specimens were charged, so that the assemblies underwent one additional heating and cooling cycle.

These assemblies were opened by abrasive cutting and examined for in-pile warping. Examination of the uranium rods revealed that the straight rod which operated at a calculated uniform temperature of approximately 550°C had warped slightly. The second straight rod which operated at a calculated uniform temperature of approximately 800°C did not appear to be warped and diameter measurements were within the original tolerance of 0.248 ± 0.001 inches.

Two tapered rods irradiated at flux levels such that one rod, Figure 28, operated at calculated temperatures varying from approximately 100°C at the large end to approximately 690°C at the tip; and the other, Figure 29, from 100°C to 900°C, did not appear to be warped. However, two pitted 1/16-inch wide circumferential bands separated by a 1/16-inch unpitted band were observed approximately 3/8-inch from the large end of the high temperature rod, Figure 29. Pits were also observed at the edge of the small end and approximately 1/2-inch from the small end.

The next step in examination of the specimens will be to polish two flat, opposing faces the entire length of each specimen. One face will be used for macro- and microetching, and the other for a series of hardness tests. It is hoped that study of the grain structure of the rods will show what portion of each rod operated in the beta phase.

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

An increasing number of fuel element failures in Hanford piles apparently arise from stress failure of the core rather than water penetration of the jacket. A failure of this type could lead to direct contact between the coolant and the hot core of the fuel element. This study will evaluate the hazard arising from the reaction between uranium at 500-750°C and water at 150-300°C.

A recirculating water loop designed for this study and intended to operate with ambient water temperatures up to 300°C and pressures up to 2000 psia was described in the previous report of this series⁽³⁵⁾. Bids have been received for off-site construction of the loop as a package unit. Delivery of the completed loop will be scheduled five months after the order is placed. Experimental work will await delivery of the loop.

Uranium Alloy Studies - M. C. Fraser

The qualitative determination of the time, temperature, transformation effects at 400°C upon gamma quenched uranium - 14 atomic percent molybdenum alloys has been extended to include temperatures of 150, 275, and 350°C.

(35) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

FIGURE 28

IRRADIATION TEMPERATURE APPROXIMATELY
100°C TO 690°C, MAG. 1.9X

Photographs Unclassified

FIGURE 29

IRRADIATION TEMPERATURE APPROXIMATELY
100°C TO 900°C, MAG. 1.5X

The uranium - 14 atomic percent molybdenum alloy specimens (hereinafter called alloy specimens) were annealed at 800°C for sixteen hours and water quenched. This heat treatment erased the existing 400°C anneal structure and reproduced the metastable gamma structure. One of the alloy specimens was examined in the as-quenched condition. Its X-ray diffraction pattern had three peaks whose 2θ angles were 36.7°, 53.0°, and 66.1° which corresponded to the (110), (200), and (211) planes of gamma uranium (72° was the upper limit of 2θ on this X-ray diffraction pattern). Its Rockwell D hardness value, an average of five determinations, was 39.

The remaining alloy specimens were sealed in evacuated quartz capsules and placed in a furnace whose temperature was 150°C. The alloy specimens were then removed from this furnace in an hourly logarithmic sequence i.e., 2, 4, 8, 16, etc., and water quenched. Each alloy specimen was then examined by X-ray diffraction and hardness measurements as before.

This heat treating technique and examination procedure was followed for the other alpha annealing temperatures of 275 and 350°C.

The results to date of this investigation are graphically shown in Figure 30. The broken line indicates the approximate times and temperatures necessary for a gamma quenched alloy specimen to begin to transform to the normal alpha plus epsilon structures.

At 400°C the structure of the gamma quenched alloy specimen underwent a significant increase in hardness within one day which was qualitatively verified by an X-ray diffraction pattern.⁽³⁶⁾ This pattern had eight diffraction maxima at different 2θ angles and only one of these peaks could be attributed to the metastable cubic gamma structure. This fact has been interpreted to mean that the relative amount of the metastable cubic structure has decreased sufficiently such that only its best X-ray diffracting plane can be identified because of its relative high intensity, i.e., the (110) plane of gamma uranium. It should be pointed out that equilibrium between the alpha and epsilon phases very likely has not been achieved in one day at 400°C but that the metastable cubic structure has been largely replaced.

A significant increase in the hardness of an alloy specimen was found after it was annealed four and two-thirds days at 350°C. In this case the X-ray diffraction pattern revealed three distinct peaks superimposed upon the characteristic cubic pattern with 2θ angles corresponding very closely with the (111), (112), and (023) planes of alpha uranium.

A similar effect occurred in nine and two-thirds days to an as-quenched alloy specimen annealed at 275°C. The hardness increase of this specimen was of the same order of magnitude as the four and two-thirds days - 350°C annealed specimen. The structural change by X-ray diffraction analysis was similar also.

An interim report is being prepared describing the in-pile test of uranium-chromium alloy specimens which were irradiated to determine the effect of

(36) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

FIGURE 30

alloying additions, per se, on the dimensional stability of uranium. The test results have been described briefly in a previous report of this series.⁽³⁷⁾ Additional exposure data have been obtained for the samples used in this test. A value of 130 MWD/AT was reported previously which was a calculated value based on records of process tube water temperatures. Due to the decreased volume of uranium in the process tubes in which these specimens were charged, the thermal neutron flux at the specimen position was higher than that at a position occupied by a normal fuel element by a factor of approximately two. Thus, the actual exposure received by the test specimen was approximately 260 MWD/T.

Literature Survey - W. E. Roake

A survey has been made of methods of returning Hanford depleted uranium to Hanford reactors. The survey report discusses (a) the thermodynamics of reduction of uranium compounds to metal, (b) processes for reduction of uranium halides and oxides to metal and to alloys, and (c) preparations of oxide ceramics.

Several areas are suggested in which experimental work appears profitable. These include (a) the effects of slag modifying agents on melting points and viscosities of bomb reduction slags with emphasis being placed on control of ultimate shape and size of uranium particles and/or coagulation thereof, (b) methods of forming high density sintered uranium oxide pieces, particulates, and cermets, and (c) development of schemes by which continuous reductions of uranium oxide to powder or pellets can be carried out.

Aluminothermic Reduction of UO_3 - W. E. Roake

Four reductions of uranium trioxide by aluminum have been carried out in the presence of sufficient cryolite, intimately mixed with the UO_3 , to dissolve the Al_2O_3 reduction product, resulting in a final slag of about 15 weight percent Al_2O_3 in cryolite. The amounts of aluminum used in the experiments were adjusted to the stoichiometric amounts necessary for theoretical reductions to alloys containing 100, 68.8, 59.5, and 52.4 percent uranium. The aluminum was melted and heated to 1050°C in a graphite crucible. The mixed cryolite-uranium oxide powder was added and the molten mass rocked slowly while soaking at 1050°C for one hour. Results are discussed in Table VI. Analysis of the metal biscuits will be made as soon as equipment is made available for melting and consolidation of the samples.

Reduction of UO_3 to Metal With Calcium - W. B. Tolley

A calcium-iodine booster with calcium as reductant has been employed for the reduction of UO_3 to the metal. The calcium iodide formed during reduction adds tremendous heat to the charge and also lowers the melting point of the calcium oxide slag. Using two and two and one-half moles of iodine-calcium booster per mole of uranium, button yields of 28.6 and 33.8 percent uranium were obtained. The uranium buttons were clean dense pieces easily broken from the slag. After leaching the slags with water and acetic acid the remaining metal powder and small spheres were collected on a filter. Over-all yields of metal recovered as regulus and powder were 97 and 94 percent respectively. It is estimated that only several percent of the uranium was pyrophoric as the bulk of the powder was easily handled during drying.

(37) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.

TABLE VI

ALUMINOTHERMIC REDUCTION OF UO_3

(For Conditions, See Text)

<u>Theoretical Product Percent U</u>	<u>Slag</u>	<u>Metal</u>	<u>Remarks</u>
100	Two layers--bottom layer black as if containing high percent of UO_2 .	No solid regulus. Metal dispersed in slag as fine particles.	Small particle analyzed 55.1% U.
68.8	Small amount black slag.	Metal in particles and solid striations throughout slag.	Microscopic examination showed metal to be porous.
59.5	Very small amount black slag.	Irregularly shaped solid chunk.	Metal will not melt at 1025°C.
52.4	Slag cream colored.	Irregularly shaped solid chunk.	Attempt to remelt metal at 1025°C resulted in small amount of metal (Al) pouring from unmelted biscuit.

Uranium Oxide Ceramics - W. E. Roake

One way in which to return Hanford depleted uranium to the piles is in the form of an oxide ceramic. Work has been started to prepare some sintered UO_2 pieces for testing as fuel elements.

Dry UO_3 , when hydrated with about 10 percent by weight of water and pressed or extruded, will set to a hard, brick-like consistency in about half an hour at room temperature. Such material has been shown in experiments at Catalytic Construction Company to maintain its integrity during hydrogen reduction to UO_2 . An approximately slug sized green compact of hydrated UO_3 has been prepared by pressing in an aluminum can at 16,800 psi for half hour at room temperature. The can was cut away from the compact which was then dried at 350°C. The drying step apparently was too rapid causing the piece to crack but not to fall apart. The piece, or another one like it, will be reduced to UO_2 and sintered as soon as facilities are available. Density of the dried green compact is 4 gm/cc.

An attempt has been made to form an approximately slug sized UO_2 compact. A mix of UO_2 , 5.25 weight percent UO_3 (as binder), and 1 weight percent water was pressed in an aluminum can for 30 minutes at 20,000 psi. The green compact will be removed from the can, and if unfractured, will be sintered. Density of the green UO_2 compact is 6.4 gm/cc.

Preparation of UF_4 - P. B. Branin, W. B. Tolley

One possible method of recycling Hanford UNH is to prepare a compound which is reducible to uranium metal suitable for slug manufacture. Some of the highly corrosive dry chemistry steps used presently in preparing UF_4 can be avoided by going to a wet method. The only practical straight chemical method known at present for reduction of UNH to U^{+4} in solution is to use ferrous ion in the presence of fluoride ion. In this case, if the fluoride ion is supplied by HF , the amorphous, hard-to-handle UF_4 precipitates. If ammonium fluoride is used instead, a dense, crystalline compound, $NH_4F \cdot UF_4$, precipitates.

This process has been carried out successfully on a laboratory scale using as a starting material about 75 g. of UNH. The UNH is dissolved in water and diluted to roughly 0.3 M. A one hundred percent excess of NH_4F is added, frequently forming a cream colored precipitate, which is dissolved by further dilution and by adjusting the solution pH to 3.3 with nitric acid. Keeping this pH quite constant, crystalline ferrous sulfate is added in one hundred percent excess, and immediately a bright green compound precipitates. The supernatant is decanted and the precipitate washed two or three times in a solution of pH 3.3. The compound has a slurry bulk density of 4 to 5, is easily filtered, and air dries to an easily handled powder.

The double salt $NH_4F \cdot UF_4$ can be decomposed by heating to $375^\circ C$ under a rough vacuum, (38) the NH_4F being trapped out, and the UF_4 remaining in an extremely finely powdered state. A batch prepared in this way has been submitted for analysis to determine whether an appreciable amount of NH_4F remains and whether there is much oxide formation. An attempt will be made to reduce this material if the analysis indicates it to be suitable.

Previous work with plutonium (39) has indicated that solid ammonium bifluoride is an excellent fluorinating agent for the dioxide at temperatures of 50 to $250^\circ C$. The resulting product is the plutonium tetrafluoride ammonium fluoride double salt. This can be decomposed to a plutonium tetrafluoride hydrate at temperatures of 300 to $450^\circ C$. Similar reactions are being investigated with uranium dioxide.

The starting material UO_2 was prepared by the reduction of UO_3 at $650^\circ C$ with hydrogen. Twenty five grams of UO_2 were reacted with 30 percent excess ammonium bifluoride over the stoichiometric amount required for the reaction.

As with plutonium the formation of the double salt is extremely rapid at temperatures of 50 to $250^\circ C$. At $150^\circ C$, apparently the optimum temperature, all the water produced during the preparation is volatilized leaving a dry powder.

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- (38) Bernhardt, H. A., Gustison, R. A., Posey, J. C., "Thermal Decomposition of the Compound Uranium Tetrafluoride-Ammonium Fluoride," K-410, June 1, 1949, p. 3, Secret.
- (39) Tolley, W. B., "The Preparation of Plutonium IV Ammonium Fluoride and Its Decomposition to Plutonium Tetrafluoride for Subsequent Reduction to Metal," HW-31211, March 15, 1954, p. 6, Secret.

During decomposition the ammonium fluoride is volatilized and collected in a cold trap while the uranium tetrafluoride remains in the reaction vessel as a powder.

Two batches were decomposed in vacuo at 375 to 400°C and a third in an argon atmosphere at the same temperature. The final tetrafluorides were dull green indicating possibly low conversion from the dioxide.

Vacuum Dry Box - D. C. Kaulitz

A vacuum dry box to provide an inert atmosphere for the safe handling of radioactive materials and other hazardous substances, such as NaK (Potassium-Sodium) has been designed and is being fabricated.

This enclosure for handling such materials is to be 4 feet long and 3 feet in diameter and to be made entirely of 3/16-inch stainless steel plate. All corners are to be rounded and the box will be of welded construction. A safety glass window extending the length of the box is to be provided at the front. An air lock for introducing equipment and materials is to be built at the right side.

The front of the enclosure is to have two 8" ports with provision for clamping rubber gauntlets to the periphery of each port. Thus, access is provided for the hands to the interior of the box for manipulation while affording protection against radiation or hazardous substances at the same time.

It is designed so that it can be completely sealed for creating a vacuum and then flushing with a controlled atmosphere of He. The box and air lock together are designed to pump to 0.1 mm of mercury and with valves closed, should not drop to less than 1 mm of mercury in 15 minutes.

To keep the atmosphere of He within the dry box free of impurities a pump has been designed that will circulate NaK over baffle plates. The He atmosphere will be circulated through this NaK curtain to remove the impurities. In order to safely operate the pump, the motor is mounted on the outside of the box and the pump driven by a magnetic clutch.

An isometric drawing showing the details of construction, such as viewing windows, gloved ports, etc., is shown in Figure 31.

The primary purpose of the unit will be for the filling of zirconium test capsules containing uranium specimens and liquid NaK. These capsules are designed for pile irradiations. Capsules have been designed for diffusion couples, tensile and impact specimens. The dry box will enable personnel to fill any size capsule without additional specialized equipment.

RADIOMETALLURGY -- L. D. Turner

Enriched Uranium From High Temperature Pile Test - D. P. O'Keefe

The enriched uranium (1.75 atomic percent U-235) slug (W624) from PT 105-532-A3 which had been irradiated at a power level calculated to generate a core temperature

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in the vicinity of the alpha beta transformation temperature⁽⁴⁰⁾ was examined to determine if any significant alteration of the metal occurred and to estimate whether temperatures in excess of 660°C existed in the slug. From a study of the metallography on a transverse section of the slug,⁽⁴¹⁾ it was concluded that the uranium at the center had been heated above the alpha beta transformation temperature. The extent of this heating throughout the slug could not be determined. There were numerous small cracks observable on a macro scale in transverse and longitudinal sections within a core of about 3/8-inch radius. Hardness testing showed an unusually large spread of 20 points on the Rockwell G scale between the center and periphery of the slug.

Normally Discharged Slug With Cracked Uranium - D. P. O'Keefe

One enriched uranium (1.75 atomic percent U-235) slug from PT 105-532-A2⁽⁴²⁾ which had a meandering indent in the can wall on one side of the slug was examined to establish the cause of the defect. On a transverse section taken near the center of the slug a fine crack, about 1/2-inch in depth, was present in the uranium beneath the indent in the jacket. The crack was closed, but apparently had been opened enough to neck down the aluminum can wall. The AlSi beneath the indent was cracked.

Ruptured Slugs From PT 313-105-19M - D. P. O'Keefe

Laboratory work was completed and a report⁽⁴³⁾ issued on Ruptures No. 279 and 283 which had been rolled and machined at FMPC. A prominent feature of Failure 279, probably related to the rolling history of the uranium, was the principal fracture plane which was on the diameter and was twisted 25 degrees throughout its length. On Failure 283, a manufacturing defect in the form of a longitudinal crack three inches long and 1/16-inch in depth, apparently existed in the uranium at the time of canning. This slug split on a diameter in the plane of the pre-existing crack.

Ruptured Slugs From PT 105-313-25M - D. P. O'Keefe, D. L. Zimmerman

Pursuant to requests by the Fuel Assembly Unit, examination of selected failures and normal slugs from PT 105-313-25M⁽⁴⁴⁾ was begun. A high failure rate has resulted in this production test of lead dip canned uranium slugs machined from salt bath heat treated Fernald rolled rods. Over half of the failures have been of the "cap failure" type in which the cap is forced from the slug.

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- (40) Lang, L. W., "Production Test 105-532-A3, Irradiation of Enriched (E-1) Uranium Slugs," HW-27850, August 1, 1953, Secret.
 - (41) O'Keefe, D. P., "Interim Report on Metallurgical Examination of Slug from PT 105-532-A3," HW-31835, (Unpublished), Secret.
 - (42) Lang, L. W., "Production Test 105-532-A2, Irradiation of Enriched Uranium Slugs," HW-27146, February 16, 1953, Secret.
 - (43) O'Keefe, D. P., "Macro-examination of Two Fernald Rolled Uranium Slug Failures," HW-31856, May 14, 1954, Secret.
 - (44) Johnson, H. A., "Production Test 105-313-25M, Lead Dip Canning and Irradiation of Uranium Slugs Machined from Salt Bath Heat Treated Fernald Rolled Rods," HW-28149, May 22, 1953, Secret.
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The examination of four ruptures of the cap failure type was begun. Exposure data and measurements on these slugs are as follows:

<u>Failure No.</u>	<u>Tube No.</u>	<u>Discharge Date</u>	<u>Canning Date</u>	<u>Tube Exp. MWD/T</u>	<u>Length at 1/2" from Axis</u>	<u>Depth of Cavity</u>
339	1988-F	4/1/54	7/27/53	518	8.354 ± 0.005 -0.011	3/16
353	3190-H	4/19/54	8/3/53	516	8.356 ± 0.010 -0.012	15/16
354	3462-H	4/21/54	7/30/53	594	8.341 ± 0.014 -0.007	3/4
362	2891-F	5/8/54	8/6/53	574	8.400 ± 0.013 -0.008	1/2

Length measurements were taken at four points about 1/2-inch from the slug axis. The above values are an average of four measurements from each slug. Since manufacturing specifications call for a length of 8.315 to 8.335, an increase in length is indicated for all slugs near the outer edge. An examination of replicas of the base end of the uranium showed all the slugs to be slightly concave on the ends. The depression in the end was perhaps as much as 0.050-inch. Deflections of this magnitude on both ends of the slug would indicate a shrinking of the total slug length at the center along with the measured increase in length at the periphery.

All of the slugs had a cavity in the uranium at the cap end, and there was very little loose corrosion product in the cavity. No cracking or other damage was seen in the uranium, an unusual circumstance considering the depth of the cavities and the possibilities for radial stresses resulting from oxide buildup in the cavity.

Three caps from PT 105-313-25M Failures No. 339, 354, and 363 were examined minutely and no evidence of material or manufacturing defect could be seen in the cap assemblies.

An examination which will parallel the work done on the ruptures was started on two irradiated PT 105-313-25M pieces which had been discharged normally. One of these pieces was selected for examination because of an indent in the can wall around the slug at the base of the cap.

Uranium Density - G. R. Mallett, L. J. Chockie

The densities of nine uranium samples were determined for the Physical Metallurgy Sub-Unit. These densities ranged from 18.93 to 18.96 gm/cm³. These samples will be irradiated on Supplement A to a production test, (45) the densities will then be redetermined to evaluate any density changes.

Examination of Flexible Connectors from 100-C - W. S. Kelly

A final report, "Examination of Flexible Connectors from 100-C," HW-31470, was issued completing the investigation of the flexible connectors from 100-C.

(45) Hueschen, R. E., "Production Test 105-3N, The Effect of Irradiation on the Properties of Uranium," HW-27831, April 24, 1953, Secret.

Examination of KEL-F Sieve Plate Material - C. L. Boyd

To aid in estimating the radiation life expectancy of the Kel-F plastic sieve plates in the various Purex columns, a number of test coupons were immersed in a HAX saturated, 2M aqueous uranium solution. Four groups of coupons were irradiated and received a total exposure ranging from 10^6 to 10^8 R. Bend and fatigue flexing tests are being conducted by the Chemical Development Unit. The irradiation produced a yellowish coloration in the plastic, but no defects or radiation damage was noticed during visual inspection. However, microscopic examination revealed two noticeable changes as a result of the immersion, namely, an increase in surface pitting and minute internal flaws or crazing. Figure 32 shows a photomicrograph of non-irradiated material, and Figures 33 and 34 of irradiated material.

A series of eight impact samples were irradiated, but the size (3" x 3/4" x 1/32" thick) made impact testing impossible.

Ultimate tensile strength tests will be conducted as soon as plastic tensile test grips are available.

Modifications to Slug Examination Cell - E. C. Watts

From experience gained in the first six months of operation, modifications to the slug examination cell were devised to improve space utilization and contamination control. Since a major portion of the examination work is performed on ruptures, an important requirement of this work is the proper control of gross amounts of contamination within the cell. To accomplish this control, the cell was partitioned with a 4" lead wall to provide two compartments 34" x 52" x 49". One compartment, in which the cleaning and primary examination of ruptures will be done (Figures 35 and 36), was fitted with a stainless steel floor which slopes to a drain at the center. A removable filter, retaining particles above 30 microns diameter, was placed in the cell drain. The filter, using glass wool as a filtering medium in a stainless steel cartridge, was designed for easy removal through a cell plug opening. A water spray, operable with the cell manipulator, was installed for the purpose of keeping the ruptured slugs wet during handling and to provide a means of washing gross contamination into the filter. An aspirator was installed in the drain line to overcome the difference in air pressure between the inside and outside of the cell and to provide some additional water flow as a carrier for the cell solutions to the crib waste system. The water supply to the wash nozzle within the cell and the aspirator in the drain were orificed to prevent excessive water flow to the crib waste system.

Equipment for viewing, photography, measuring, jacket removal, and storage can handling was installed on the cell walls so that the floor can be kept clean and free of contamination traps. Wherever possible, such equipment is installed on removable cell plugs.

Access between the two compartments, through the lead partition, was provided by a three-inch access port. The remaining compartment (Figure 37) was provided with a drain to crib waste and a Hanford slave manipulator installed. This compartment can be kept free of gross contamination and is available for tests on cleaned slugs and other materials requiring the shielding afforded by the high level cell.

FIGURE 32

NON-IRRADIATED - MAG. 150X

FIGURE 33

IMMERSION TIME 56 HOURS,
EXPOSURE 10^7 R - MAG. 150X

FIGURE 34

IMMERSION TIME 560 HOURS,
EXPOSURE 10^8 R - MAG. 150X

Photographs Unclassified

SLUG EXAMINATION CELL RADIO METALLURGY BLDG.

FIGURE 35

Photographs Unclassified

FIGURE 36

NORTH COMPARTMENT OF SLUG EXAMINATION CELL

Photographs Unclassified

FIGURE 37

SOUTH COMPARTMENT OF SLUG EXAMINATION CELL
(NOTE REMOVABLE PALLET AND ACCESS PORT
THROUGH PARTITION)

Cut-Off Machine - C. L. Boyd

The abrasive cut-off machine^(46,47,48) has been placed in operation and a number of successful cuts have been made on irradiated slugs and production test containers.

Eight wafers from an enriched uranium slug and one wafer from a normal uranium slug were sectioned and shipped to the General Physics and Chemistry and Chemical Engineering Units at KAPL.

The first successful longitudinal sectioning of a Hanford fuel element was accomplished when two longitudinal cuts were made on a two-inch long portion of slug number W-624 from PT 105-532-A3.⁽⁴⁹⁾

Metallographic Cell - C. L. Boyd

The metallographic cell^(46,47) has been placed in operation and a number of samples prepared for X-ray diffraction studies and metallography.

Tensile Machine Modification - W. S. Kelly

The modification of the Tinius Olson tensile machine to permit the testing of irradiated tensile samples at room temperature was completed. Tensile tests on four samples of irradiated uranium with an average dosage rate in air of 350 R/hr at 17 inches was completed without overexposure of operating personnel.

High Temperature Tensile Test - W. S. Kelly

The tentative design of a unit which can be used to determine the tensile properties of irradiated uranium at temperatures up to 700°C has been completed.

The vacuum chamber in which the samples will be placed during testing, containing the vacuum seals through which the shafts of the tensile grips must operate, has been fabricated. The unit is being prepared for testing and, if a suitable vacuum is attained to prevent excessive oxidation of uranium at elevated temperatures, the other necessary equipment such as a high temperature furnace and special extensometer equipment will be built.

In-Cell Vacuum Annealing Furnace - W. S. Kelly

The in-cell vacuum annealing furnace⁽⁴⁸⁾ was successfully used to anneal two irradiated uranium samples at 400°C for 15 hours at a vacuum of $\sim 5 \times 10^{-5}$ mm of mercury. A report is being issued describing the unit.

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- (46) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," HW-30114, January 25, 1954, Secret.
 - (47) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - October, November, December, 1953," HW-30896, March 5, 1954, Secret.
 - (48) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954," HW-31799, May 10, 1954, Secret.
 - (49) Lang, L. W., "Production Test 105-532-A3, Irradiation of Enriched (E-1) Uranium Slugs," HW-27850, August 1, 1953, Secret.
- - - - -

Bend Testing of Irradiated Uranium - W. S. Kelly

A bend test jig was designed and is being built to permit tests to determine the physical properties of samples cut from whole slugs. Bend testing of irradiated uranium, while possibly not as valuable as tensile testing, will provide a measure of the strength and ductility. Bend test samples, being more readily formed than tensile samples, could be cut from any desired slug using the abrasive cut-off wheel. Standard shaped tensile samples cannot be prepared at the present time.

Macroetching - G. R. Mallett

A closed box and air scrubber to remove acid fumes during the macroetching of irradiated uranium has been developed and is being installed for obtaining satisfactory macrostructures by chemical and electro-chemical means.

Electropolisher - R. E. Field, C. L. Boyd

A manually operated electropolisher, which will allow samples to be gripped and lowered into several electrolytic and cleaning tanks, is being designed for use on irradiated samples.

To insure adequate cleaning of sample between electropolishing and cleaning stations an ultrasonic generator and transducer elements have been requisitioned to be installed in each of these tanks.

Cathodic Etching - R. E. Field, G. R. Mallett

Modification of the vacuum cathodic etcher located in the Radiometallurgy Building is being conducted to permit remote etching of irradiated samples. Successful etching of non irradiated uranium has been completed as reported by T. K. Bierlein elsewhere in this report and by Ames Laboratory.⁽⁵⁰⁾ Experience gained and methods used in non-irradiated sample etching will be incorporated into the remote operated etcher as much as possible.

The remote portion of the unit will consist of a glass discharge tube, located within the cell, containing an aluminum anode and a water-cooled zirconium cathode on which the specimen will be placed. Remote controls will be provided to lower the cathode from the unit to permit sample loading and unloading. Suitable controls located outside the cell will permit the tube to be evacuated, krypton to be introduced at 50 microns pressure and a maximum potential difference of 5000 volts D.C. at a maximum of 50 milliamps to be applied between electrodes.

Rockwell Hardness Tester - R. E. Field, C. L. Boyd

A remotely operated sample positioner, for use on the existing Wilson Rockwell hardness tester, was fabricated and installed. (See Figure 38.) Pneumatic operated jaws grip the sample which may then be positioned under the indenters by means of two 27 V-DC motor driven slides. Hardness readings may be taken in increments of 0.015-inch across the face of a sample.

(50) Carlson, A. J., Williams, J. T., Rogers, B. A., Manthos, E. J., "Etching Metals by Ionic Bombardment," ISC-480, May 30, 1954, Unclassified.

FIGURE 38
REMOTELY OPERATED SAMPLE POSITIONER
INSTALLED ON ROCKWELL HARDNESS TESTER

A Bodine motor drive was also added to the table with final vertical positioning being manually controlled to insure accurate adjustment of initial load.

Sample Punching - R. E. Field

A hydraulic press, punch and die assembly to punch small cylindrical samples from slug wafers is being designed for use in the cells of the Radiometallurgy Building.

The press will be operated by means of a 20,000 pound vertically mounted hydraulic cylinder, attached to the upper portion of the press, and will have a maximum stroke of four inches. T-slots located on the bed of the press will permit die assemblies to be bolted rigidly to the press frame.

A punch holder, screwed to the hydraulic ram, will accommodate punches ranging in size from 1/16-inch to 1/4-inch diameter. To prevent clinging of the wafer to the punch on the upstroke a spring loaded stripper plate has been incorporated into the design. The die assembly clamps the wafer and allows accurate rectilinear positioning of the wafer beneath the punch. Removable die buttons and punches will provide a wide selection of sample sizes. Manual controls for positioning wafer and for remote removal of punches and die buttons will be located outside of the cell.

Carbon Analysis - G. R. Mallett

The equipment for obtaining a carbon analysis on irradiated materials has been completed. The furnace and sample dumper have been made and are ready to be assembled.

Scintillation Sample Positioner - R. E. Field

In support of a program to determine the geometrical distribution of the fission products in Hanford fuel elements, a slug wafer positioner is being fabricated for use in the cells of the Radiometallurgy Building. (See Figure 39.)

This unit will allow a thin slug wafer to be translated uniformly past a small hole located in one of the cell plugs. A gamma spectrometer, located on the outside face of the plug, will be used to analyze the fission product distribution across selected diameters of the wafer.

One and one-half-inch diameter wafers, up to 3/16-inch thick, may be loaded into the positioner using a manipulator. The wafer rests on rollers which may be rotated to position the wafer for any chosen scanning diameter. The wafer is then positioned in front of an 1/8-inch hole in the cell plug and by means of a rack and pinion drive translated past the hole.

Thermal Conductivity - R. E. Field, C. L. Boyd

A remote operated unit for accurately determining the thermal conductivity of irradiated samples is being designed.

One end of the sample will be heated, by a combustion tube furnace, to a maximum temperature of 1000°C, while the other end is being water-cooled. Thermocouples, spotwelded to the sample, will provide temperature readings along the surface of the sample. A silvered heat shield surrounding the sample will also be heated

FIGURE 39

SCINTILLATION SAMPLE POSITIONER

and cooled in a similar manner to that of the sample to prevent radial heat flow from the sample. The entire unit will be enclosed within a vacuum system to prevent oxidation of the sample during the furnace heating cycle.

The sample will be introduced and withdrawn from the unit by means of a hydraulic ram.

Electrical Resistivity - C. L. Boyd, R. E. Field

The electrical resistivity measuring apparatus⁽⁵¹⁾ shown schematically in Figure 40 is capable of measuring cylindrical and rectangular samples 1/16-inch to 1/2-inch wide and 3/4-inch to 6 inches long. The entire unit is submerged in a tank containing a constant temperature oil bath having a maximum temperature of 450°F. The control rods pass through high temperature silicone rubber seals located in the tank wall and then through the cell wall. The distance between electrodes is adjustable and may be measured accurately by means of a rod extending through the cell wall and actuating a dial indicator. This arrangement permits the distance between electrodes to be measured within ± 0.001 -inches. An electric stirrer mounted on the tank lid provides for circulation of the oil bath and maintains a uniform temperature gradient across the tank.

Density Cell - G. R. Mallett

The density equipment has been altered to provide greater accuracy and easier operation than the Mettler balance unit.⁽⁵²⁾ The balance has been placed above the shielding, and enlarged and better controlled storage and cleaning areas have been incorporated. The liquids used to measure density can be regulated to $\pm 0.05^\circ\text{C}$ by a constant temperature bath. The equipment that is shielded is arranged as shown in Figure 41. This present arrangement allows for easier manipulation of samples and for smoother operation in cleaning and weighing radioactive samples. Calibration of the balance and system have yet to be done to insure good accuracy.

WELDING AND CORROSION - W. R. Smith

Corrosive Effects of Cr^{VI} in Nitric Acid and ANN - K. L. Sanborn

Data previously reported⁽⁵³⁾ were indicative of the marked increases in corrosion rates to be expected of stainless steels 304L and 309SCb when exposed in nitric acid solutions to which Cr^{VI} ion had been added. The data, however, were reported as average corrosion rates based on the accumulated weight losses for varying numbers of 48-hour boiling periods because in many cases the corrosion was so severe the tests had to be discontinued in order to preserve the specimen. In the course of evaluating the consistency of the data it was noted that if log corrosion rate determined from the first 48-hour boiling period were plotted against log Cr^{VI} concentration, that a family of relatively straight lines resulted for the various nitric acid concentrations investigated. These curves have rather steep slopes indicating that the effect of Cr^{VI} is pronounced, and are displaced toward higher corrosion rates as the nitric acid concentration is increased. The curves are presented in Figure 42.

- (51) Kemper, R. S., Boyd, C. L., "Electrical Resistivity Measurement of an Irradiated Uranium Sample," HW-28309, June 8, 1953, Secret.
- (52) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - January, February, March, 1954" HW-31799, May 10, 1954, Secret.
- (53) Cadwell, J. J., "Quarterly Progress Report - Metallurgy Unit - July, August, September, 1953," HW-30114, January 25, 1954, Secret.

Photographs Unclassified

FIGURE 40

FIGURE 41

THE EFFECT OF CR VI ON THE INITIAL CORROSION RATE OF TYPE 304L STAINLESS STEEL IN BOILING NITRIC ACID AND ALUMINUM NITRATE NONAHYDRATE SOLUTIONS.

FIGURE 42

The curves have little practical importance for the corrosion rates are, for the most part, in ranges which would rule out the possibility of a successful application of stainless steel. Further, the rates were calculated on the basis of only 48 hours exposure time in a deliberate effort to eliminate the factor of grain dropping and, therefore, are considerably lower than would be experienced in actual service. They do show the effect of Cr^{VI} on the tendency for the stainless steel to react with Cr^{VI} bearing nitric acid, and it is for this purpose only that they are reproduced here.

The apparent consistency of the data for nitric acid solutions prompted similar tests in 70% aluminum nitrate nonahydrate (ANN), the principal constituent of the Redox waste. As shown on the plot in Figure 42 the curve for 70% ANN appears below that for 10% nitric acid and at a lesser slope.

While the practical significance of these curves is small, they are recorded for reference in the analysis of problems involving chromium effects.

Redox Waste Storage Tank Corrosion Test - N. Endow, G. R. Mallett

Some results of a field corrosion study of SAE 1020 carbon steel exposed to Redox process waste solution for a period of nine months have recently become available. The interest in corrosion data for this material in neutralized Redox waste solution stems from its use as the liner component of the 75-foot diameter tanks used in storing these highly radioactive wastes. The purpose of the present field test is to obtain rates of general attack, of pitting and local type attack, and of possible stress corrosion cracking of steel exposed to the liquid-sludge phase of the waste media.

The temperature of this waste was in excess of 300°F during a considerable portion of this test. From the test, it was learned that the extent of pitting attack after extended exposure (nine months) is not serious. The deepest pit observed was approximately 0.003 inches deep. If one assumes that the pitting rate were constant over the entire exposure period, the rate of this type attack would be approximately 0.0003 inches per month. The uniform corrosion rates for steel exposed to the waste show a maximum of 0.00001 ipm (inches penetration per month). No cracks were observed at 2X and at 200X magnification on the single corrosion test specimen which was stressed to near the yield point. This specimen is at present being sectioned for metallographic examination.

Redox Waste Concentration Corrosion Studies - N. Endow

Laboratory corrosion tests of types 304L and Carpenter 20 stainless steel and of commercially pure titanium have been continued in both the present Phase I and in the proposed Phase II Redox D-12 Waste Concentrator bottoms. These tests are being conducted for ten exposure periods of 48 hours each in the solution at boiling temperatures at atmospheric pressure and also at elevated temperatures corresponding to heating coil skin temperatures in sealed ampoules under pressure. Type 304L stainless steel is being tested as representative of the best type of stainless steel available for this concentrator heat exchanger service and of the material presently used, while TI-75A is being tested as a material which shows promise in laboratory tests to date and which might be considered as an alternative for this service. Titanium weld specimens made by butt welding titanium were included in the tests. Metallographic examination of these welds prior to testing

indicated that sound titanium welds were being tested. Also, the present series included tests designed to determine the effect of adding sodium nitrate to the Phase II waste solution as a means of reducing the hexavalent chromium present. Previously, the efficiency of this treatment in Phase I solution at 120°C was shown. The results of all of these tests to date are given in Table VII; they show that the Phase II solution used in this series of tests is not as corrosive to type 304L stainless steel as Phase I solution when exposures are carried beyond the 5th period; however, the corrosion rate is excessive in either case. These results are in contradiction with expected results inasmuch as the Phase II solution has 2.3 times as much hexavalent chromium as the Phase I solution. The difference may be due to the use of the same Phase II solution after the 5th period whereas fresh aliquots were used in the case of the Phase I solution.

The titanium has shown excellent corrosion resistance to the Phase II solution, as well as to the Phase I solution, the data for which was reported previously. This is true for both the parent metal and for titanium weld metal. Titanium samples exposed continuously for 240 hours in Phase I D-12 solution at 166°C also exhibited excellent corrosion resistance. One defect was noted on the specimen pre-treated with HCl at 20°C; this specimen had a small smooth pit covered by a small deposit, the composition of which has not been determined. Examination of other titanium specimens which have not been exposed revealed the presence of similar defects.

Tests in the reduced Phase II D-12 solution show that at 120°C type 304L stainless steel is not severely attacked even when exposed for longer than 240 hours. The data from the 70% ANN test indicate that the ANN component of the waste solution is not the principal corrodent at 120°C.

Pilot Plant Heat Exchanger Studies - N. D. Groves, G. W. Riedeman

The most serious corrosion problem existing in the Hanford separations plants today is what material of construction and what methods of fabrication should be used in constructing process heat exchangers. To date all of the major failures due to corrosion in the Hanford separations plants have been in the heat transfer portions of heat exchanger vessels.

In an effort to learn more about the effect of heat transfer duty on the corrosion rate of various materials in some of the process solutions, a mock up concentrating vessel--the F-4 pot, 321 Building--has been constructed and operated in a manner simulating process conditions. This vessel contains five separate heat exchangers which allow variations in materials and fabricating techniques to be evaluated simultaneously.

The first set of heat exchangers comprised two types of heat exchangers fabricated from types of 304L, 347, 309SCb and Alloy #20 stainless steels. These heat exchangers are completely described in HW-31799. The medium of exposure was simulated Redox D-12 waste concentrate and the operating conditions involved 41 days at 32 psig and 53 days at 80 psig steam pressures. These tests are qualitative in nature but very clearly indicate the following facts.

1. The corrosive attack is greatly accelerated in the areas where heat transfer takes place.

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TABLE VII
CORROSION RATES FOR STAINLESS STEELS AND TITANIUM
IN PRESENT AND MODIFIED REDOX WASTE SOLUTIONS

<u>Solution</u>	<u>Metal</u>	<u>Average Corrosion Rate First Five 48-Hour Periods (ipm)</u>	<u>Per.No.</u>	<u>48-Hour Period Corrosion Rate for Given Corr.Rate(ipm)</u>
Phase I	SS, 304L	0.0031	8	0.0139
D-12 Solution	SS, 304L	0.0029	8	0.0077
120°C	SS, Carp.20	0.0084	8	0.0119
	SS, Carp.20	0.0085	8	0.0114
Phase II	SS, 304L	0.0018	10	0.0028
D-12 Solution	Ti, 75-A	0.00003	10	0.00001
120°C	Ti, 75-A			
	one side butt-welded	0.00001	8	0.00001
	Ti, 75-A			
	both sides butt-welded	0.00001	8	0.00001
Phase II	SS, 304L	0.0005	9	0.0006
D-12 Solution	Ti, 75-A	0.00002	9	0.00001
Cr ⁺⁶ reduced with NaNO ₂ , 120°C				
70% Aluminum Nitrate	SS, 304L	0.00008	9	0.00009
Nonahydrate Solution	Ti, 75-A	0.00001	9	0.00001
120°C				
Phase I	Ti, 75-A			
D-12 Solution	HNO ₃ Treated	0.00001		
165°C	Ti, 75-A			
	HCl Treated	0.00001		

(Continued from page 88):

2. There is no appreciable difference in corrosion rate between the cannister type and the bayonet type heat exchanger.
3. The grades of stainless steel tested are all unsuitable for heat transfer duty in Redox waste concentrate. However, of the four types of stainless steel tested, type 304L appears to offer the most resistance to corrosive attack.
4. Tungsten inert gas shielded welds made with type 308L welding alloy on all of the base materials tested appear to be more resistant to corrosive attack than the base materials.

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The second series of heat exchangers have been built and went into service on June 13, 1954. Analysis of the first set of exposed heat exchangers reveals that the greatest degree of corrosion occurred in the alloy #20 which has the highest nickel to chromium ratio and that the degree of attack descends in the same order of alloys as the nickel to chromium ratio's decrease. Two new methods of forming tubing have shown some advantages and improvement over the conventional processes. The glass lubricated extrusion process produces a seamless tube with improved surfaces which are practically free of microfissures. The "Rockrite" process may provide large diameter pipe with improved surface characteristics. The current set of specimens are designed to further evaluate these and other design and material variables.

There are five units now in service; three are of the cannister type and two are of the bayonet type. The materials involved in the cannisters are:

OUTER SHELLINNER SHELL

3" Sch. 40 Extruded Type 347

Special Welded 1" Tube

3" Sch. 10 Weld-drawn Type 304L

1" Sch. 40 Weld-drawn 329

3" Sch. 40 Rockrite Type 304L

1" Sch. 40 Extruded 304L

One outer shell has a 2" x 3" type 312 patch welded into the wall with type 312 welding rod. One bayonet exchanger consists of four 1" tubes under test. Two of these tubes consist of several short lengths of pipe welded with different alloys, one of which has been solution heat treated at 2050°F for one hour. The third and fourth tubes are type 304L extruded and type 329 weld-drawn pipe. The remaining bayonet exchanger is a special single tube unit in which a 1" Sch. 10 weld-drawn titanium tube is under test. Because of material limitations, the lower test section is the only part fabricated from titanium. The titanium tube is flanged and bolted to the stainless steel supply and support members. The titanium specimen is made such that a fusion weld of two different designs as well as a crevice condition exist.

The current exposure is being made in simulated Redox waste concentrate with 85 psig steam pressure on all of the heat exchangers. It is planned that this experiment will continue for three months or until a failure occurs, whichever is the shorter time.

Bearing Studies - N. D. Groves

The continued search for materials which are suitable for use as process stream lubricated bearings has lead to the evaluation of several new products.

Tungsten Carbide - Specimens of grades C-93 and 101 Talide (tungsten carbide) were obtained from Metal Carbides Corporation who reported the following literature characteristics of the material:

	<u>Grade C-93</u>	<u>Grade 101</u>
Composition		
percent Tungsten Carbide	94	98
percent Cobalt	6	2
Hardness, Rockwell "C"	78-79	83-84
Density, g/cc	14.85	15.40

Samples of these two materials were exposed to boiling 65% nitric acid as a screening test. Both specimens developed a tightly adherent film of yellow corrosion product which is believed to be tungsten trioxide. X-ray identification of the corrosion product was attempted but the lack of a suitable standard prevented positive identification. The corrosive attack appeared to be uniform and the rates observed are indicated in Table VIII. Since these rates are considered excessive in themselves and would probably be increased in a flowing system the investigation of these materials has been discontinued.

V2B Alloy - V2B is a hardenable 18-8 stainless alloy containing copper, molybdenum, silicon, and a very small amount of beryllium produced by the Cooper Alloy Foundry Company. In the quench annealed condition this alloy is soft enough to be readily machined but after machining the steel may be precipitation-hardened by a low temperature heat treatment which produces no distortion. This alloy is reported to be non-galling and very hard.

As a comparative screening test, specimens of V2B which had been subjected to a variety of heat treatments were exposed to boiling 65% nitric acid for five 48-hour periods and the corrosion rates calculated from weight loss measurements. The heat treatments that were examined are:

Sample No.

1 & 2	Sensitize one hour at 1250°F, water quench
3 & 4	2100°F for one hour, water quench
5 & 6	2100°F for one hour, water quench, then precipitation harden for 8 hours at 925°F and furnace cool.
7 & 8	Precipitation harden the "as received" sample for 8 hours at 925°F and furnace cool.
9 & 10	The "as received" condition.

The extent of corrosion calculated as uniform penetration in inches penetration per month is shown in Table VIII.

Based on the fact that all of the corrosion rates were excessive and also the fact that samples Nos. 1, 2, and 5 cracked radially during the exposure, investigation of this alloy has been discontinued.

Tantung - Specimens of Tantung alloy produced by the Vascoloy-Ramet Corporation have been subjected to boiling 65% nitric acid and boiling 40% nitric acid. Tantung alloy has a nominal chemical composition of 50% cobalt, 30% chromium, 10-15% tungsten, with the balance being carbides.

The tests in boiling 65% nitric acid were stopped after 48 hours because of the high rate of corrosion. The tests in boiling 40% nitric acid are being continued since the first period rate was not considered conclusive for rejection. These rates are tabulated in Table VIII.

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TABLE VIII
CORROSION RATES OF VARIOUS BEARING MATERIALS

<u>Material</u>	<u>Specimen No.</u>	<u>Exposure Media</u>	<u>Exposure Time (Hours)</u>	<u>Corrosion Rate (ipm)</u>
Tungsten Carbide	C-93	Boiling 65% HNO ₃	48	0.0036
		"	96	0.0042
	101	"	48	0.0037
		"	120	0.0049
V2B	1	"	240	0.0895
	2	"	240	0.0905
	3	"	240	0.0239
	4	"	240	0.0372
	5	"	240	0.0518
	6	"	240	0.0446
	7	"	240	0.0446
	8	"	240	0.0506
	9			0.0508
	10	"	240	0.0516
Tantung	301-1	"	48	0.0369
	301-2	"	48	0.0783
	301-3	Boiling 40% HNO ₃	48	0.0270
	301-4	"	48	0.0168
Carbolube AP-80	PL	Boiling 65% HNO ₃	500	0.651%*
Carbolube AP-80 w/Sb	AN	"	48	Dissolved

* Percent weight loss

Carbolube - Specimens of carbolube (fused graphite), grade AP-80 and grade AP-80 impregnated with antimony, produced by Carbone Corporation, have been exposed in boiling 65% nitric acid for a maximum of 500 hours. The grade AP-80 impregnated with antimony dissolved within 48 hours but the plain grade AP-80 appeared to withstand the attack of boiling 65% nitric acid for 500 hours. The specimen suffered a 0.6% weight loss during the 500 hours but did not suffer from crumbling or softening. These data are tabulated in Table VIII.

Specimens of both grades are currently being exposed to 40% nitric acid at 140°F.

Examination of Tube Bundles for the Redox Waste Concentrator (D-12) and Oxidizer (H-4) - W. R. Smith, K. L. Sanborn

Seven bayonet type tube bundles have been fabricated on-site for service in the Redox waste concentrator (D-12) and/or oxidizer (H-4). Three have been fabricated by maintenance forces and four by Minor Construction. The tube bundles were fabricated using type 304 stainless steel pierced drawn pipe for tubes. The use of this material made it mandatory, following welding, that the tube bundles be given a solution heat treatment followed by a water quench to prevent intergranular corrosion in the heat affected zones adjacent to the welds.

All the tube bundles exhibited evidence of intergranular corrosion attack on some of the tubes adjacent to the tube sheet from the short exposure in the pickling solution and all the tube bundles suffered distortion and warpage from the heat treatment. The four tube bundles fabricated by Minor Construction were welded with type 347 rods and electrodes. Many of the welds were cracked and many of the metal-arc welds contained slag inclusions and voids. The three tube bundles fabricated by maintenance forces were welded with type 308L rods and electrodes and all the welds appeared to be free of defects except about ten welds on the face of one tube sheet. An investigation showed that these defective welds were the result of poor workmanship.

It is considered that all of these tube bundles contain defects which may shorten their service life.

Welding Tests of High Strength Low Alloy Steels, Project CG-558 - G. W. Riedeman

Three low alloy high strength steels, Mayari-R, Yaloy, and Cor-Ten, have been tested to determine their weldability under the conditions similar to those that would exist during the actual fabrication of the replacement downcomers at 100-B and 100-D. Normally these steels require a preheat to be successfully and reproducibly welded, but in this application such a procedure appears to be impractical. Consequently, the determination of the weldability of these steels without a preheat was the object of this study.

The plate materials were cut into test plates and welded, making a final plate about 10" x 15" with a 60° "V" joint welded in the center in the 15" dimension. The face and root bend, tensile, and metallographic specimens were then cut from one end of the plate and the remaining plate was loaded as a single beam and subjected to a longitudinal weld face bend. The object of this test was to determine if cracks that develop in the weld will propagate into and through the base material. If the base material fails, the type of fracture indicates if