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QUARTERLY PROGRESS REPORT
FUELS DEVELOPMENT OPERATION
JANUARY, FEBRUARY, MARCH, 1961

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April 14, 1961

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Work performed under Contract No. AT(45-1)-1350 between the
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The present Quarterly Report is the continuation of a series issued by the new Fuels Development Operation. Reports in this series combine portions of the quarterly reports by the former Metallurgy Research and Fuel Technology Sub-Sections. Work reported includes research conducted by the Physical Metallurgy Operation, and research and development conducted by Fuel Design, Fuels Fabrication Development and Ceramic Fuels Development Operations. Studies formerly reported by the Radio-metallurgy, Metallography, and Welding and Corrosion Units, in addition to portions of the Fuels Technology work, are reported elsewhere.

Reports previously issued as Metallurgy Research and Fuel Technology Quarterlies are listed in the first report of the present series, HW-46091.

QUARTERLY PROGRESS REPORTS - FUELS DEVELOPMENT

Reports previously issued are as follows:

HW-46091	July, August, September, 1956.
HW-47850	October, November, December, 1956.
HW-49803	January, February, March, 1957.
HW-51704	April, May, June, 1957.
HW-53488	July, August, September, 1957.
HW-54702	October, November, December, 1957.
HW-56029	January, February, March, 1958.
HW-57146	April, May, June, 1958.
HW-58205	July, August, September, 1958.
HW-59235	October, November, December, 1958.
HW-60237	January, February, March, 1959.
HW-62085	April, May, June, 1959.
HW-62656	July, August, September, 1959.
HW-64863	October, November, December, 1959.
HW-66094	January, February, March, 1960.
HW-67904	April, May, June, 1960.
HW-69085	July, August, September, 1960.
HW-70355	October, November, December, 1960.

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SUMMARYPHYSICAL METALLURGYMECHANICAL AND PHYSICAL PROPERTIES OF IRRADIATED MATERIALS

Recovery studies on irradiated iron have indicated the strong dependence of the recovery process on impurity atoms.

The yield point observed in irradiated type "A" nickel specimens is apparently dependent on a simple diffusion process enhanced by radiation induced defects.

Zircaloy-3 has retained a few per cent uniform plastic elongation after irradiation to 10^{20} nvt.

The powder metallurgy aluminum alloy M-257 shows no change in tensile properties with irradiation while aluminum alloy X-8001 exhibits a small increase in strength accompanied by a gradual loss in ductility.

Three second generation capsules for measuring creep in-reactor are being modified to improve their in-reactor operating characteristics.

The activation energy for the ex-reactor creep of Zircaloy-2 has been determined to be multi-valued over the temperature range from 55-400 C. A sharp peak in the activation energy is observed at about 270 C.

MECHANICAL AND PHYSICAL PROPERTIES OF UNIRRADIATED MATERIALS

The pole figures determined on uranium spheres and hemispheres cut from rods fabricated by several techniques have been interpreted according to suggested deformation mechanisms.

FUNDAMENTAL STUDIES

The irradiation of various evaporated films of Pd, Pt, and Ge in contact with UO_2 has shown that the recoil fission fragment damage is a function of the film thickness.

It has been established that dislocation movement in irradiated aluminum foils is impeded by fission fragment damage regions.

Pre- and post-irradiation X-ray diffraction measurements on annealed and cold worked aluminum foils have indicated a slight increase in lattice parameter after neutron exposure.

Three controlled temperature general swelling capsules have completed their irradiation and two additional capsules have been charged into the reactor. These latter are operating at constant temperatures of 525 and 575 C, respectively.

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Samples of NPR inner tube and NPR outer tube given a variety of holding temperatures and quenching rates showed that dimensional changes and grain size were nearly constant regardless of the heat treatment used. A marked columnar grain structure has been observed at higher quench rates.

Simulated dual enriched rod elements have been fabricated with a significant reduction in area in the uranium of the outer component prior to coextrusion. The interface roughness of this rod was approximately 0.003 inch as compared to 0.008 inch in rod made from cast and heat treated components.

FUEL EVALUATION

Irradiation of 1.75-inch OD by 1.06-inch ID Zircaloy-2 clad tubular fuel elements to exposures of 1200 and 2000 MWD/T has been completed. Irradiation performance was good and post-irradiation measurements of dimensional changes and fuel element swelling have been completed.

Eight 20-inch KER-size tube-tube fuel elements have been irradiated to 2000 MWD/T. The outer tubes contained unbonded closures and the inner tubes hot headed and projection welded closures. Destructive examination reveals no significant deterioration of either closure. Slight bumping was evident on both inner and outer tubes.

Post-irradiation examination of tube-tube fuel elements irradiated to 3200 MWD/T was continued. Dimensional changes and metallography of a uranium 2 w/o zirconium fueled tube has been completed.

Ten 15-inch NPR inner tubes, processed with variable beta heat treatment, were irradiated to 1050 MWD/T. The elements will be dimensionally characterized to determine the effects of variation in heat treatment.

Fuel elements with closures formed using an 84 w/o Zircaloy-2, 4 w/o Be, 12 w/o Fe braze have been irradiated in a single pass facility in the MTR and the 6x9 recirculating loop in the ETR. The cold water test failed at 1050 MWD/T. A cladding rupture was evident at the base of the brazed heat-affected zone. The elements irradiated in the 6x9 loop operated satisfactorily. The outer tube operated to an exposure of 1200 MWD/t and the inner tubes operated to an exposure of 825 MWD/T. Destructive examination has been initiated on both sets of elements.

Defect testing of irradiated coextruded Zircaloy-2 clad uranium rods continues to show an increasing rate of corrosion damage with increased exposure. However, the irradiated fuel corrosion rate is greatly reduced when the loop water temperature is decreased to temperatures below 200 C.

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Thermal Conductivity

Measurements were completed on an irradiated and a nonirradiated UO_2 specimen.

High Temperature Microscopy

Equipment for high temperature microscopy at elevated pressures was placed in operation.

Fuel-Cladding Reactions

High temperature compatibility studies of UO_2 and UC with various cladding materials were initiated. Tantalum was the most compatible with UC, and tungsten was the most compatible with UO_2 .

Tubular UO_2 Fuel Element Irradiation

A vibrationally compacted fused UO_2 , Zircaloy clad, tubular element was irradiated in a high temperature, high pressure test facility.

Nested Tubular Fuel Element Irradiation

A prototypic nested tubular fuel element was fabricated by vibrational compaction for an irradiation test. The Zircaloy clad element contained high energy impact-formed UO_2 .

Recycled UO_2 Irradiation

Previously irradiated UO_2 was partially decontaminated by the "Salt Cycle" process of the CR&D operation, fabricated, and assembled into a cluster by remote techniques, and successfully re-irradiated.

PHYSICAL METALLURGYMECHANICAL AND PHYSICAL PROPERTIES OF IRRADIATED MATERIALSEffects of Irradiation on Nonfissionable Materials - J. J. Laidler and K. R. Wheeler

Neutron Damage to Metals. The purpose of this program is to advance the theory of irradiation damage to metals by neutrons. Copper, nickel, titanium, zirconium, iron, molybdenum, and type 347 stainless steel specimens were irradiated at Brookhaven, Hanford, and the MTR under various exposure and temperature conditions. Post-irradiation measurements of mechanical and physical properties which were initiated at KAPL will be completed at HAPO.

The iron specimens used in this program originated from three ingots which varied slightly in interstitial impurity content. Ingot 1 contained 100 ppm total interstitial impurities, while ingots 2 and 3 contained 170 ppm and 230 ppm, respectively. The results of a series of isochronal anneals on specimens from each of the three ingots are shown in Figures 3.1 and 3.2. While microhardness measurements are relatively insensitive to subtle changes, the effect of increasing interstitial impurity content is still evidenced by a difference in damage recovery. Figure 3.2 shows that recovery of electrical resistance occurs in three stages for all impurity levels. The temperature of maximum recovery rate for each comparable recovery stage increases with increasing impurity content, with the exception of the first stage, which occurs in all cases at 175 C. Isothermal annealing of Ingot 1 specimen at 175 C has shown this reaction to be characterized by first-order kinetics, with an activation energy of 39,000 cal/mole (based on microhardness measurements). This value is considerably higher than the activation energies reported for diffusion of C in α -Fe. The possibility that small areas of the matrix have transformed to the γ structure is not out of the question, since similar observations have been made on cold worked iron, (1) with an activation energy for carbon diffusion of 33,000 cal/mol.

Figure 3.3 shows recovery curves for iron specimens of three exposure levels. It is quite interesting that only one stage, that at 240-260 C, exists for more than one exposure. This suggests that this stage is related to basic material parameters rather than to radiation-induced defects, the distribution of which is believed to be constantly changing with increasing exposure. The reaction which occurs at this temperature range also results in the suppression of the yield point observed in tensile tests, further indicating that the process must involve impurity atoms. A possible mechanism is resolution of carbon or nitrogen due to the action of thermal and/or displacement spikes and precipitation in the temperature range 240-260 C.

Further evidence of the marked effect of impurity level upon damage accumulation in iron is shown in Figure 3.4, where the increase in lower yield stress is shown as a function of exposure for specimens from ingots 1, 2, and 3. The method of Petch⁽²⁾ was used to separate radiation hardening into

FIGURE 3.2

Isochronal Recovery of Electrical Resistance of Irradiated Iron

FIGURE 3.4
Radiation Hardening in Iron

FIGURE 3.5
Lattice Hardening (σ_1) in Iron as a Function of Fast Neutron Exposure

Preliminary data from another group indicated the possibility that Zircaloy-3 is sensitive to strain rate. However, upon examining subsequent results and in view of the small number of specimens initially tested, the difference in data at the two strain rates could be attributed to experimental scattering. Additional tests are planned to clarify these observations. Two observations can be made regarding a stress-elongation plot of irradiated Zircaloy-3 as illustrated in Figure 3.7. Unlike Zircaloy-2, this alloy has retained a few per cent of uniform plastic strain at these exposure levels. Also, the damage effects seem to be saturating at that particular irradiation temperature (approximately 90 C), and exposure (10^{20} nvt). Because of their corrosion resistance in certain media and low absorption cross section aluminum alloys are used extensively in some reactor applications. The aluminum powder metallurgy alloy M-257 is of interest because of its superior high temperature strength as compared to conventional aluminum alloys. A high nickel aluminum alloy, X-8001, has good corrosion resistance in high temperature water. Figure 3.9 illustrates graphically the change in stress and strain for X-8001 and M-257 resulting from irradiation. For X-8001 the change is a small gain in strength with gradual ductility loss. M-257, however, shows no appreciable radiation induced changes due to the nature of its structure. The effects of oxide dispersion in its matrix influence dislocation movements to a much greater extent than do irradiation defects.

In-Reactor Measurements - L. J. Chockie, J. J. Holmes, and J. C. Tobin

The properties of structural materials are altered both by neutron damage and the dynamic effects of fast neutron irradiation. The objective of the in-reactor measurements program is to define the physical and mechanical behavior of structural materials during fast neutron irradiation. Currently a study of the in-reactor creep properties of Zircaloy-2 is being conducted. A prototype in-reactor creep test capsule has been successfully operated for more than 10,000 hours. Preliminary data from the prototype capsule show that the creep of Zircaloy-2 is greatly increased in a fast neutron flux. In order to further investigate the in-reactor creep properties of Zircaloy-2, four additional capsules of modified design were purchased. Capsule II-4 of the new series or second generation capsules was charged in the K-West reactor in December, but unexpected high gamma heating and a weakness in the thermocouples resulted in the ultimate failure of this capsule as a creep testing instrument. Capsule II-4 will, however, be used to test the in-reactor operation of a mechanical extensometer and to check the validity of creep strain measured with undamaged sections of the two three-range transducers. (Fuels Development Operation Quarterly Report, October, November, December 1960)

Capsule and Instrument Development. On the basis of the in-reactor operation of Capsule II-4 it was decided to modify the remaining three capsules before irradiation. Modification was conducted at the vendor's plant where emphasis was given to reworking one of the capsules to provide an early shipping date. Prior to modification Capsule II-2 was thermal cycled

FIGURE 3.8

Stress-Strain Behavior of Irradiated Aluminum Alloys M-257, X-8001

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FIGURE 3.9

Modified Second Generation Creep Capsule

REC-GE RICHARD WASH

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FIGURE 3.10
Spectrum of Activation Energies for the Creep of Zircaloy-2

ROD P

FAB. HISTORY: Extruded at 620 C at 13:1 extrusion ratio.
Swaged from 0.593-inch diameter to 0.500 inch -
diameter at room temperature, ~ 30 per cent
reduction in area.

POLE FIGURE SPECIMEN: 1/4-inch diameter hemisphere
with 1/4-inch diameter shank. Shank axis is also
the swaging axis.

MICROSTRUCTURE: 100 X

FIGURE 3.12

Experimental Data, Rod P

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ROD A

FAB. HISTORY: Sylvania random powder metallurgy compact.

POLE FIGURE SPECIMENS: 3 geometries to match those shown
for Rods N, P, and L.

MICROSTRUCTURE

100 X

500 X

FIGURE 3.14

Experimental Data for Rod A

FIGURE 3.15

Pole Figures 020 and 110 of α -Extruded Uranium Swaged at Room Temperature to 12.5 Per Cent Reduction in Area

ROD N

FIGURE 3.17

Pole Figure 111 of α -Extruded Uranium Swaged at
Room Temperature to 12.5 Per Cent Reduction in Area

FUNDAMENTAL STUDIES

Optical and Electron Microscopy - T. K. Bierlein, B. Mastel, and H. E. Kissinger

Effects of Irradiation on Thin Films. Electron microscopy with its high resolving power permits direct examination of defects such as dislocation loops and fission fragment damage in thin films and foils. The extent of radiation damage in various materials can be determined by comparing pre- and post-irradiation states. This information in turn permits a better understanding of the basic mechanisms of radiation damage.

Studies of fission fragment damage in evaporated metallic and non-metallic films of the type carbon-X- UO_2 have continued. In these films, X refers to Cr, Ni, Pd, and Be, and the films contain discrete regions which are free of UO_2 .⁽⁶⁾ It is in such regions that recoil fission fragment damage is observed. Damage is found to be similar to that observed in previously reported films, in which X = Al, Ge, Pt, ZrO_2 , and C. If tracks are observed directly by transmission microscopy, they are seen by virtue of a contrast effect due to changes in the amount of scattering material along the path taken by the fission fragment. In the case of materials having low electron scattering power such as evaporated Be, C, and Al, contrast differences are very slight and fission damage is not seen as readily as in films containing material having high electron scattering power. If the irradiated films are shadowed to reveal topographical changes, all materials reveal similar characteristics. A series of mounds form on the surface of the material X, which is free of UO_2 , and these mounds lie adjacent to a trench which formed as a result of removal of X atoms due to transfer of energy from the fission fragment.

Since control and measurement of evaporated film thicknesses is difficult, a series of films with a constant, known ratio of film thicknesses was prepared. A number of 1/8-inch diameter grids with an approximately 200 Å thick carbon substrate obtained from a single evaporation were coated during a subsequent single evaporation with approximately 80 Å of UO_2 . A series of these double layered films, all of equal thicknesses, was placed on a stepped wedge having predetermined distances between steps. Since the thickness of an evaporated film is inversely proportional to the square of the distance between film and source, it is relatively simple to satisfy the condition that film thicknesses on successive steps on the wedge have a ratio of 1:2. With this fixed thickness ratio, films of Pt (4.5, 6.75, 9, 18, and 36 Å), Pd (27, 40, 54, 108, and 216 Å), and Ge (92, 138, 184, 368, and 736 Å) were prepared and irradiated to 3×10^{16} nvt (thermal). Comparison of tracks in any particular series of films which differ only in the thickness of metal X shows differences in track contrast and geometry, Figure 3.19. Tracks in all films are characterized by discrete circular regions which are considerably more transparent to electrons, and can almost be considered as holes through the composite film. It is quite evident that in the case of the thinner Pd layers, extension of the damage region adjacent to the high

damage regions occurs, and that such damage becomes less apparent as the thickness of the Pd layer increases. In the thinnest film, most tracks, especially the shorter ones, show that essentially all material is removed at one end of the track (presence of holes) whereas the other end generally shows less electron transmittancy. An attempt to evaporate additional Pd onto the irradiated films containing incremental thicknesses of Pd, so that final film thickness differences would be eliminated, was made. Although photometric measurements indicated essentially equal thicknesses among the recoated irradiated films, differences in contrast effects at tracks still persisted. The significance of this is that damage to films must indeed be a function of thickness and is not simply due to an artificial contrast effect due to instrument parameters or to differences in electron scattering through layers of different thicknesses. An additional factor associated with fission damage in thin films having incremental thicknesses has been established. During irradiation in an air environment (the possibility of chemical reactions with the gaseous environment must not be ignored), particles with high electron scattering decorate the free surface along a fission track, and they are most prevalent and apparent in the intermediate thickness range. In the case of the C-UO₂-Pd films, the Pd thickness is 40 Å. This has been further substantiated in shadowed C-UO₂-Pt films for which the Pt thickness for maximum decoration of tracks with electron opaque particles was 6.75 Å.

If a fission fragment with a given energy suffers discrete energy losses as it passes through a metal, since the matrix metal atoms affected by the fission fragment behave differently depending on their distance from the free surface, it may be possible to ascribe a direction of travel to the fission fragment. In a previous report, HW-70355 (October, November, December 1960 Quarterly), it was shown that the top and bottom surfaces of a film are affected differently. Track geometry, namely the tapering of tracks, their width, and the extent of damage or holes associated with the two ends of a track, may also provide a basis for determining track direction. The study of fission fragment registration and characterization in thin, evaporated films is continuing.

Fission fragment tracks in aluminum foils are similar to those in evaporated films, HW-70355 (October, November, December, 1960 Quarterly). In order to determine what type of interaction occurs between a moving dislocation and a fission fragment damage region in aluminum, additional studies have been made. As the sequential micrographs in Figure 3.20 illustrate, dislocation motion appears to stop when the dislocation approaches a fission fragment damage region. A complication in the direct examination of foils containing fission tracks and dislocations arises. For maximum contrast of dislocations, contrast of a grain containing a fission track is generally low, and fission tracks are, therefore, less perceptible. Although fission fragment damage in a bulk specimen may be considered to be much less severe than in a thin foil specimen, one must bear in mind that an interaction may still occur. It is hoped that further studies will show whether such interactions or pinning of dislocation by fission fragment damage regions is an important factor in the hardening of fuel and cladding materials.

Irradiation of annealed and cold worked aluminum foils, 0.003 inch and 0.001 inch in thickness, have continued. After an exposure in a Hanford Snout Facility to 1×10^{19} nvt (thermal), the foils were thinned and examined in the microscope. Both the annealed and cold worked foils disclosed the presence of a small number of dislocation loops, approximately 300 Å in diameter. The cold worked specimen showed the highest concentration, Figure 3.21. Since no such loops were detected in similar, unirradiated foils, and since they resemble loops observed in aluminum quenched from high temperatures, it can be concluded that irradiation has resulted in vacancy clusters which after collapse form the small dislocation loops. On a qualitative basis motion of dislocations in the irradiated foils appeared to be impaired compared to the ease of dislocation movements in nonirradiated control specimens. Since the dislocations after irradiation appear to contain a large number of jogs and frequently exist as tangles, additional energy may be required for equivalent movements. Moreover, interaction between collapsed vacancies, the small dislocation loops, and normal dislocation lines may also contribute to the decrease in the tendency for dislocations to move in the irradiated foils. The sequential micrographs of a specimen which had been annealed prior to irradiation, Figure 3.22, do illustrate that pinning of a dislocation line at a small loop may occur. As was the case in the study of interaction between dislocation lines and fission fragment damage regions, in general, a compromise orientation which permits observation of both dislocation line and the small loops simultaneously results in a decrease in optimum contrast obtainable when each defect is examined individually. Similar studies on more highly irradiated aluminum foils are in progress.

X-ray diffraction was employed to measure the lattice parameters of the Al samples before and after irradiation. Values of the lattice parameter, a_0 , were obtained by measuring the d-spacings for four high angle lines, 331, 420, 422, and 333. The values of lattice parameter derived from these measurements were plotted against $\cos^2 \theta$, and extrapolated to $\theta = 90^\circ$. Copper K α irradiation, $\lambda (\alpha_1) = 1.54051 \text{ Å}$, $\lambda (\alpha_2) = 1.54433 \text{ Å}$ ⁽⁷⁾ was used. The values of a_0 are:

Annealed, pre-irradiation	$a_0 = 4.04860 \text{ Å}$
Annealed, irradiated 10^{18} nvt fast	4.04900
Annealed, irradiated 10^{19} nvt fast	4.04900
Cold worked, pre-irradiation	4.04882
Cold worked, irradiated 10^{19} nvt fast	4.04902
All values are $\pm 0.00005 \text{ Å}$	

Broadening of the X-ray lines was very slight. For the annealed samples, differences in breadth between the irradiated samples and the unirradiated samples were of the same order as those between individual unirradiated samples. Inasmuch as the annealed foils contained a very small number of crystallites suitably oriented to diffract, differences between individuals were pronounced. The lines for the cold worked samples were broader than those for the annealed samples, but were not further broadened upon irradiation.

FIGURE 3.22

Sequence Micrographs of Jogged Dislocation Tangles and Small Loops in Annealed Aluminum Which Was Irradiated to 1×10^{19} nvt (Thermal). The Motion of a Dislocation Line Which Passes Near a Loop is Arrested. Magnification of Negative Print is 80,000X

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To provide a sound basis for evaluating the changes that occur in uranium during irradiation at elevated temperature, it was felt that a series of laboratory capsules should be annealed so that they have the same thermal histories as in-reactor capsules. Five laboratory capsules have been completely assembled and a vacuum annealing system has been modified to handle the capsules. Each laboratory capsule contains one specimen identical to its counterpart that has been irradiated or is being irradiated. Comparisons will then be made to determine the differences that result with respect to geometry, microhardness, density and microstructure.

Two unmonitored NaK filled capsules, each containing a precharacterized U-U diffusion couple specimen, are under irradiation in the MTR in a flux of 5×10^{13} nv. The calculated maximum temperature of the uranium is < 200 C.

Pore Size Distributions. Optical and electron microscopy afford a direct means for detecting accumulated fission gases in irradiated uranium and obtaining gas pore size distributions. Such information is needed in order to understand how gas atoms migrate, coalesce, and grow into large pores. Replicas of uranium with burnups of 0.29 a/o and 0.41 a/o after a variety of post-irradiation annealing treatments are being analyzed by quantitative metallographic techniques, and the changes in pore frequency distributions as a function of thermal treatment are being ascertained.

Specimens cut from Zircaloy-2 and stainless steel clad rods of uranium with burnups of 0.29 a/o and 0.41 a/o, respectively, were de-clad and annealed for times of one hour and 100 hours at temperatures of 400, 500, 600, 650, 700 and 880 C. The low annealing temperatures were included because changes in the pore distributions at the higher temperatures were so very large. For example, a one-hour anneal at 880 C of a specimen having a burnup of 0.29 a/o caused a change in density from 18.62 g/cc to 12.77 g/cc after annealing. Pores in this specimen are so large and their distribution so heterogeneous that quantitative metallography is all but impossible.

Table 3.2 lists pertinent swelling data obtained with specimens having 0.29 per cent burnup and various post-irradiation annealing treatments. Data from both buoyancy density measurements and quantitative metallography is included. Swelling is best indicated by the change in volume of the specimen divided by the initial volume, $\frac{\Delta V}{V_i}$, but since the volumes were determined by density measurements, the numerically equivalent $\frac{\Delta \rho}{\rho_f}$ (the change in density divided by the final density) values are reported. On the other hand, quantitative metallography leads to $\frac{\Delta V}{V_f}$ values since the statistical treatment of measured pore diameters yields a pore void fraction which is the ratio of the volume of pores measured to the volume of uranium plus the volume of all existing pores. As this ratio should be numerically equal to $\frac{\Delta \rho}{\rho_i}$, both pore void fractions, VF in Table 3.2, and $\frac{\Delta \rho}{\rho_i}$ values are included.

The swelling data obtained from density determinations for 0.29 a/o BU samples annealed for 100 hours and for 0.41 a/o BU specimens annealed for 1 and 100 hours is summarized in Table 3.3. As the statistical treatment of the micrographs of these specimens is not yet complete only $\frac{\Delta \rho}{\rho_f}$ and "R" values are reported. "R" is simply defined as $\frac{\Delta V}{V_i} \times 100$ divided by the per cent burnup. This provides a convenient way for comparing the data from specimens having different burnup. It can be argued that swelling is not a linear function of the burnup as implied in the definition of "R" but these values still provide a better basis for comparison than do simple volume changes. It is interesting to note in Table 3.2 and 3.3 that there is no apparent time dependence of swelling. This may be somewhat misleading because of the slow heating and cooling rate employed but qualitatively, at least, the data points out the extreme temperature dependence of swelling compared with the time dependence.

TABLE 3.3
POST-IRRADIATION SWELLING OF URANIUM

<u>Specimen History</u>	<u>ρ g/cc</u>	<u>$\frac{\Delta \rho}{\rho_{final}}$</u>	<u>"R"</u>
Initial State	18.90		
As-Irradiated, 0.29 a/o	18.56	0.0183	6.30
400 C, 100 hrs	18.54	0.0194	6.69
500 C, 100 hrs	18.39	0.0277	9.55
600 C, 100 hrs	17.26	0.0950	32.7
650 C, 100 hrs	18.14	0.0418	14.4
700 C, 100 hrs	17.18	0.1000	34.5
880 C, 100 hrs	17.38	0.0874	30.1
Initial State	18.92		
As-Irradiated, 0.41 a/o	18.70	0.0118	2.88
400 C, 1 hr	18.53	0.0210	5.12
500 C, 1 hr	18.03	0.0493	12.0
600 C, 1 hr	16.11	0.1742	42.5
650 C, 1 hr	15.35	0.2325	56.7
700 C, 1 hr	15.25	0.2408	58.7
880 C, 1 hr	14.73	0.2845	69.5
400 C, 100 hrs	18.50	0.0227	5.54
500 C, 100 hrs	17.84	0.0605	14.75
600 C, 100 hrs	14.97	0.2640	64.4
650 C, 100 hrs	14.95	0.2655	64.7
700 C, 100 hrs	15.20	0.2445	59.7
880 C, 100 hrs	14.63	0.2930	71.5

$$R = \frac{\Delta \rho}{\rho_{final}} \times 100 / (\text{per cent burnup})$$

FIGURE 3.23 A

Pore Volume Versus Pore Diameter Distribution Curves

Pore VF/Unit Cell Width, W_1 , from Curve, $\times 10^{-5}$		Pore VF per Cell, $\times 10^3$		No. of Pores/cc of Specimen per Cell, $\times 10^{-10}$	
3.05	9.82	0.482	1.459	67.62	252.47
3.47	3.10	0.622	0.521	59.85	60.18
4.10	3.80	0.837	0.727	53.68	56.78
4.55	4.35	1.058	0.948	46.29	50.33
4.92	7.78	1.309	1.930	38.84	69.47
5.25	5.10	1.577	1.449	30.93	34.94
5.40	5.39	1.869	1.750	20.76	23.70
4.99	5.25	1.973	1.930	14.63	18.01
3.79	4.35	1.699	1.820	8.52	11.30
2.60	3.19	1.387	1.528	4.59	6.22
1.35	1.90	0.790	1.038	1.75	2.73
0.80	1.00	0.530	0.617	0.66	1.23
0.43	0.60	0.326	0.421	0.38	0.49
0.18	0.29	0.155	0.234	0.09	0.24
0.03	0.09	0.029	0.083	0.01	0.05
	0.01		0.010		0.00
Total		101.09	31.09	936.7	

Gas Atoms/cc of Specimen: 6.1×10^{19}

Center Core Region
500 C Anneal, 1 Hour
0.28 a/o Burnup
 $\Delta \rho / \rho_{\text{initial}} = 0.0338$
VF = 0.028 (0.031)
VD = 8.13×10^{13} /cc of Specimen
VF / VD = 3.2×10^{-15} cc/Pore

FIGURE 3.23C

Pore Volume Versus Pore Diameter Distribution Curves

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FIGURE 3.23 E

Pore Volume Versus Pore Diameter Distribution Curves

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yields a value for total number of gas atoms, N , contained in a cc of the specimen, i. e.

$$N = \sum_i n_i = \frac{K}{T} \sum_i \frac{VF_i}{r_i} .$$

From a consideration of the measurement technique employed, it can be shown that

$$N = \frac{4.7 \times 10^{23}}{T} \sum_i VF/\text{unit cell width}.$$

Figure 3.23 includes values for N calculated on the basis of the above relationship.

Since the swelling induced in the specimens may be altered due to the heating and cooling rates used, difference distribution curves are listed in Figure 3.24. Such curves should show changes in pore void fraction distributions due to holding at temperature, because the swelling associated with the heating and cooling cycles are essentially cancelled. Attempts at utilizing a single parameter for representing a given distribution of pores have not succeeded in establishing how gas atoms add to existing pores and cause growth of pores. Qualitative conclusions derived from the curves in Figures 3.23 and 3.24 definitely indicate that: (1) at low annealing temperatures, below 500 C, only small pores are growing in size, and they do not grow larger than 3000 A; (2) since agglomeration of observable small pores into larger pores would result in a decrease in the number of small pores, and since an increase in the number of observable pores actually occurs, pores much smaller than the lower limit, 800 A in diameter, must be growing; (3) as the shapes of the distribution curves indicate, a very large number of very small, nonobservable pores must exist, and since the probability of agglomeration is a function of the actual number of pores present, as well as their size, changes in pore size distributions are determined primarily by the large number of very small pores; (4) after annealing at temperatures higher than 500 C, an increase in the number of large pores, 3000 A to 9000 A, results and this is accompanied by a decrease in number of small pores, 800 to 3000 A in diameter. The change in distributions caused by the high temperature of annealing can, therefore, be explained on the basis that agglomeration again occurs principally between pores of essentially equal sizes, but these pores are bigger by virtue of what can occur at the lower temperatures or during the preceding stages of pore growth.

If fission gas atoms are truly insoluble in the matrix uranium, the number of gas atoms associated with pores should be independent of the state of the specimen. As already indicated, a large number of nonobservable pores contain gas atoms, and a deviation from the theoretical number can be expected. This deviation, however, should decrease with higher annealing temperatures of a specimen, which is the case.

$$\text{Cell Number} = \frac{1}{r} (\ln \text{Pore Diameter}_{\text{obs.}} - S)$$

(r and s are constants)

Derived from Figures 3.23 D Minus C

$$\text{Cell Number} = \frac{1}{r} (\ln \text{Pore Diameter}_{\text{obs.}} - S)$$

(r and s are constants)

Derived from Figures 3.23 E Minus D

FIGURE 3.24 (contd.)

Difference Distribution Curves

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That agglomeration of pores does occur and must be one of the important growth processes is indicated by micrographs which show dumb-bell shaped pores. In addition, very large pores seen in high burnup uranium (0.41 a/o) after high temperatures of annealing frequently show a very non-spherical geometry with small spherical protuberances or pores. In the latter case, the minimization of surface area has presumably not been complete. Various models for pore growths are being devised and checked with the pore size distribution data which has been obtained to date.

Fission Product Mobility. A knowledge of the inert fission gas mobilities in uranium is important to the understanding of the basic mechanism of swelling. Several approaches are currently being pursued in an effort to establish this mobility. The first involves studying uranium specimens that have had inert gas introduced into the surface by "glow discharge" or ion bombardment. The gas release characteristics are being determined and the microstructure is being examined for gas pore formation. The second approach involves the study of U-U diffusion couples with microhardness and metallographic traverses across the U-U interface. One portion of the diffusion couple will contain an appreciable quantity of fission products while the other will contain little or no fission products.

Two 1/2-inch long by 1/2-inch diameter disks cut from a copper clad (unbonded) extrusion having a core of depleted uranium (0.15 per cent U^{235}) and an 0.030-inch shell of enriched uranium (3 per cent U^{235}) are currently being irradiated at ambient temperature in the MTR in a flux of about 5×10^{13} neutrons per square centimeter per second. One surface of each disk was metallographically polished, cathodic vacuum etched, marked with Knoop hardness indents and, finally, replicated. The surface will be again replicated after irradiation so that identical areas can be compared before and after irradiation. The goal exposure in the enriched shell is 0.3 a/oBU. The change in pore size distribution across the depleted-enriched interface as a function of post-irradiation annealing will be studied by quantitative metallography. If measurable changes occur, reasonable estimates of the fission product diffusion coefficients should be possible. This type of experiment eliminates the concern over the diffusion barrier that is unavoidably present at the surface in the "release" type of experiments which have been conducted to date with irradiated uranium in order to determine the fission gas mobility.

Continued study of the gas release characteristics of the glow discharge system previously reported has indicated that the system itself contributed the major portion of the krypton that was evolved. The uranium disk was removed, the system re-evacuated and then heated. When the previous maximum temperature was exceeded, krypton was once more evolved. This could have been released from the glass walls, the uranium that had condensed on the walls or from the cathode components which were nickel and tantalum. It is likely that the uranium evaporated onto the walls was mainly responsible. In order to better evaluate the amount of krypton that was actually in the specimen, it was thus necessary to construct a new heating system separate from

Radiometallurgy examination of nineteen uranium fuel rods irradiated in NaK Swelling capsules in a Hanford reactor is complete except for some chemical burn-up analysis. Several of these rods have cladding splits and severe bulging which prevented making complete dimensional measurements on all the clad rods. An analysis of the dimensional and density measurements, temperatures, and burn-up is being made and the results will be presented in the next quarterly of this series.

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METALLIC FUEL DEVELOPMENT

NEW FUELS DEVELOPMENT

The Effects of Cladding Thickness Uniformity on Zircaloy-2 Clad Coextruded Rods - J. W. Weber

Failures of Zircaloy-2 clad uranium rods and tubes as a result of localized clad straining have occurred in NaK filled capsules and high temperature recirculating water loop irradiations.

Metallography on one sample, rerun of chemical burnup analysis on three samples, and density measurements on three samples is all that remains to be done on the radiometallurgy examination of 19 Zircaloy-2 clad rods irradiated in NaK capsules in a Hanford reactor. The first chemical burnup analysis indicated the burnup to be 25 to 50 per cent higher than calculated. If this is true, the calculated temperatures would also have to be increased. Calculation of circumferential cladding strains from the measured pre- and post-irradiation diameters of the rods irradiated to 2000 MWD/T has been completed. Those rods with 0.020-inch cladding showed from 1.5 to 2.0 per cent cladding strain and those with 0.030-inch cladding all showed less than 1.4 per cent cladding strain. The 0.020-inch clad rods all suffered ductile failures or localized plastic strain (necking) in the cladding. None of the 0.030-inch clad rods at this exposure showed indications of localized plastic strain; however, one rod incurred a brittle clad failure. The strain on this failed cladding is less than 0.4 per cent. Circumferential strain in 0.020-inch and 0.030-inch clad rods irradiated to 1100 MWD/T have the same relative values as found at the higher exposure, that is, the strain of the 0.020-inch clad rod is greater than for the 0.030-inch. Measurements of the cladding thickness on a striated 0.020-inch clad rod showed the striation occurred where the cladding thickness changed from 0.022 inch average to 0.013 inch within a 0.050-inch gage length. There were several other locations where the cladding was 0.015-0.016 inch thick, but the thickness changes were gradual and extended over a considerably greater distance. The 0.030 inch clad was found to have equally as great thickness variations but the percentage thickness change was considerably less than for the 0.020-inch rod. It would appear then that an appreciable percentage thickness change over a short gage length may be necessary to initiate local clad instability. To verify this observation, additional macro pictures have been taken of striated and unstriated rods of both 0.030- and 0.020-inch cladding from which cladding thicknesses can be measured.

To further study the effects of cladding thickness variations on the susceptibility to failure, a series of NaK capsule irradiations of Zircaloy-2 clad fuel rods is planned for the Hanford reactors. Eight unalloyed rods have been coextruded and fuel rod samples with no striations and with

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FIGURE 4.1

Axial Elastic Strain in Cladding as Determined by Measuring the Height of a Two-Inch Long Section Before and After Dissolving Away the Uranium Core

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Fuel Element Deformation Studies - K. R. Merckx

An initial study⁽¹⁾ of a fuel element model for tubular fuel elements accounted for the resistance of the fuel material to shearing stresses but did not allow for the cyclic straining in the cladding material. For fuel materials with low resistance to shearing stresses, this study indicated that the resistance of the fuel material could be neglected; hence a fuel element model assuming hydrostatic stresses in the fuel material has been formulated. A material model for the cyclic straining in the cladding material is proposed. Incremental straining each cycle is accounted for in the material model. Assuming a power relation between the strain ranges and maximum stresses, the resulting equations for tubular fuel elements are evaluated for the ratios of interior cladding strain to exterior cladding strain and of exterior cladding strain to irradiation dilatation. If $S = AE/E^n$, where S = maximum stress intensity, A = parameter, n = parameter, E = strain range intensity, then the above ratios of strains are predicted⁽¹⁾ to be:

$$\frac{\delta e_i}{\delta e_o} = \left(\frac{x}{t}\right)^{1/1-n} = \frac{\text{inner circumferential strain increment}}{\text{outer circumferential strain increment}}$$

and

$$\frac{\delta e_o}{\delta \theta} = \frac{1-x^2}{2\left[x^2 \frac{x}{t}^{1/1-n} + 1\right]} = \frac{\text{outer circumferential strain increment}}{\text{irradiation dilatation increment}}$$

where

t = interior cladding thickness/ exterior cladding thickness

x = inner fuel radius/ outer fuel radius.

In order to evaluate parameters and test the validity of the material model for the cladding, a testing frame and loading mechanism has been designed and fabrication has begun. A strain gage extensometer has been built. The testing apparatus can control extensions in both tension and compression and the stresses in the specimen are measured.

Increased Strain Rates Due to Large Temperature Cycles - K. R. Merckx

Polycrystalline uranium under cyclic thermal conditions develops internal stresses which increase strain rates under constant loads. Previously in this series of reports, test results were reported for specimens temperature cycled between 150 C and 450 C. Specimens loaded to 1000 and 2000 psi were cycled between 150 C and 350 C. In order to use these results on models for fuel element behavior, the mechanical strains occurring during heating and cooling needed to be differentiated.

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Several closures fabricated by the "self-brazing closure" process (formerly known as the "extrusion closure" process) were checked with the new testing device. It was revealed that the closures were not consistently sound, but it appears that increasing the thickness of the copper plate on the cap tends toward improved bonding. Three out of 14 self-brazed closures tested were entirely sound in the cap/clad bond; the remainder exhibited varying degrees of soundness.

As an experiment, three of the closures with defective bonds were treated by placing them in a copper collet on the lower electrode of a large electric spot welder; the upper electrode was placed in contact with the end face, and with a load of about 1200 pounds, a few pulses of heavy current were passed through the closure zone. This resulted in heating the closure zone sufficiently to cause a light oxide film to form on the jacket surface. Where this film was uniform, retesting in the Damping Test indicated that the poor bonding had been repaired. Thus it appears that the consistency of the process is largely dependent on the control of heating and pressing conditions. Facilities directed toward these ends are being fabricated for experimental trials.

Projection Welded Resistance Braze Closure - P. A. Ard and W. I. Steinkamp

The projection welded resistance brazed closure developed on Zircaloy-2 clad coextruded rods was attempted on Zircaloy-2 clad (1.050-inch OD by 0.500-inch ID) tubes with encouraging results. This closure is being evaluated as a possible alternate method of closing coextruded Zircaloy-2 clad uranium fuel elements. The closure consists essentially of projection welding a Zircaloy-2 cap to the element cladding followed by a fast resistance heating of the cap to braze the cap to the uranium surface. The uranium is first recessed approximately 0.025 inches to provide a cladding projection ring to match a projection machined in the closure cap. A cross-section of the closure on a tubular element is shown in Figure 4.3.

The work on the tubular elements was performed on a 100 kva spot welding machine operating at full capacity. Results indicate that this closure is feasible on tubular elements, however additional power and pressure will be necessary to produce a high quality reproducible projection weld which is necessary for this type of closure. The welding force was not sufficient to prevent the weld zone from blowing out occasionally during the projection weld. An increase of welding pressure would require an increase of welding power which was not available on the 100 kva machine to fuse the projections. A cross-section of a projection weld on a tubular element showing the tendency toward blowing out is shown in Figure 4.4.

A copper chemical immersion plate was plated on the cap to aid in bonding the cap to the uranium surface. A cross-section of an interface between the cap and the uranium of a tube is shown in Figure 4.5.

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FIGURE 4.5

Cross-Section of Interface Between Zircaloy-2 Cap and Uranium on
1.050-Inch OD by 0.500-Inch ID Tubular Element. Copper Immersion
Plate Used as Bonding Agent

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welding apparatus and by the operation of the turntable used to rotate the element as it is being welded. Any variation in the wire feed, or a voltage fluctuation, will produce an unacceptable weld as will a variation in turntable speed or a fuel element which is out of alignment in the chuck by as little as 1/32 inch.

Again, the step cut is a major factor in controlling the amount of braze in the weld bead. By placing the welding arc as far from the braze ring as possible, the initial heat from the weld is also concentrated at a point away from the braze. The arc penetration is also away from the braze, which would ideally cause the molten pure Zircaloy-2 to only just flow over the braze area. The step width, then, must be a compromise between trying to get the welding heat and penetration as far away from the braze as possible, and the width necessary for a good weld profile. At the present time, a step width of 1/8 inch is being used with a step height of 3/64 inch.

The width of the braze also is a factor in securing a braze free weld. The wider the braze, the more braze will disperse through the weld. A maximum braze width of 0.015 inch is suggested with a width of 0.010 inch or below as ideal.

Welding voltage is directly proportional to the amount of heat of the weld and thus to the amount of braze in the weld. Machine settings of between 18 and 20 volts have produced almost braze free welds if all other conditions were right. At the present time, machine settings of 17 and 18 volts are being used and are expected to produce good results.

All welds to the present time have been made on the outside braze ring because of the inability of the apparatus to make a satisfactory weld on the inside rings. For this reason, a step was machined on the OD of a pure Zircaloy-2 rod and a weld bead made. The piece was then placed in an autoclave to see if the weld would pass this test. No corrosion appeared on the specimen after the autoclave cycle was completed indicating the welding procedure acceptable thus far.

Steel Clad Zircaloy-2 Self Supports for NPR Outer Fuel Tubes -
J. P. Pilger

Prototypical steel clad Zircaloy supports on the dummy element in the Elmo-7 facility, as of March 18, have been exposed for 2098 hours to "ex-reactor" water. Loop water conditions are 300 C at 2000 psi at pH 10. No visible detrimental effects had occurred to the supports at that time. The elements were recharged and continued in this environment. The loop had been emptied and decontaminated during the 2088 hour exposure. No crud buildup was observed in the support clearances after restartup following decontamination, as had been previously observed on steel clad supports of an earlier design.

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but the power required is somewhat greater. Figure 4.9 shows a weld made with a single impulse of two cycles duration with the machine set for three phase operation. The uranium has been affected beneath the weld zone. This has not been shown to be detrimental in reactor operation but appears to be a possible source of swelling or other undesirable behavior. With three phase operation and a 4.4 volt secondary, the heat affected zone in the uranium could be minimized but could not be entirely eliminated.

TABLE 4.1

0.030-INCH ZIRCALLOY SUPPORTS RESISTANCE SPOT WELDED
TO 0.020 INCH CLAD COEXTRUDED ZIRCALLOY STOCK

<u>Electrode Radius</u> <u>(Inches)</u>	<u>Electrode Force</u> <u>(Pounds)</u>	<u>Phase Shift</u> <u>(Per Cent)</u>	<u>Average Shear</u> <u>Strength (Pounds)</u>
4	50	20	1050
4	75	15	950
4	75	20	1100
4	75	25	1035*
4	75	30	1100**
4	100	20	1000
4	150	20	980
4	150	25	1010
2	150	26	980
2	150	35	1250

Fixed Conditions

Welder = Sciaky 100 kva, three phase, used, single phase with increased voltage (3.8 volts).

Weld time = one impulse of one cycle.

Welds made singly using spring loaded electrode holder.

All electrodes class 1 material.

* See Figure 4.6

** See Figure 4.7

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50 X, Polarized Light

FIGURE 4.7

0.030-Inch Support Resistance Spot Welded to 0.020-Inch Clad Coextruded Zircaloy Stock; 30 Per Cent Phase Shift, Single Phase 8.8 Volts Secondary, 75-Pound Electrode Force.

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50 X, Polarized Light

FIGURE 4. 9

0. 030-Inch Support Resistance Spot Welded to 0. 020-Inch Clad Coextruded
Zircaloy Stock; 37 Per Cent Phase Shift, 3 Phase, 2 Cycle, 4. 4 Volts
Secondary, 300 Pound Electrode Force.

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FIGURE 4.10

Heating Curve for Uranium Billet
(11 Inches in Diameter by 11 Inches in Length) in Liquid Heat 980 at 725 C.

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FIGURE 4. 12

Ingot Number 33258T - As Cast

1/2 X

FIGURE 4. 14

Ingot Number 33258T
After Third Beta Heat Treatment.
Bottom Surface

FIGURE 4. 16

Macrostructure of Primary Extrusion U-38.
Notch Indicates Top of Die

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Heat Treatment Studies - R. N. Johnson

Sixteen samples of NPR inner tube 59 (30-mil inner clad, 38-mil outer clad, 1.263 OD, 0.431 ID) and 16 samples of NPR outer tube 74 (25-mil inner and outer clad, 2.401 OD, 1.740 ID) were given a variety of heat treatments consisting of beta temperatures of 710, 730, and 740 C, air delay times before quenching of 20 seconds, and quench into nitrate salt at 250 and 300 C. The purpose of the heat treatments was to determine the degree of reproducibility of dimensional changes, cooling rates, grain sizes, and growth indices that can be obtained in any one beta treatment and to determine the effect of variations of beta treating temperature and quench temperature on the final product. In addition, five samples of NPR outer tube were given a beta treatment in NuSal at 730 C, 20 second air delay before quenching, and quenched into Houghton quench "G" oil at room temperature for comparison with those quenched in the nitrate bath. Good reproducibility in dimensional changes, cooling rates, and grain sizes was found in each heat treatment. In fact, dimensional changes and grain sizes were nearly constant regardless of the heat treatment used, indicating that the heating and quenching temperature is not too critical, at least as far as these two properties are concerned. Grain sizes of the NPR inner tube samples were consistently A2 to A3 and the grain sizes of the NPR outer tube samples were consistently A5 to A6 (a finer grain size). Preferred orientation was determined by X-ray examination in three directions (transverse, longitudinal, and radial), and the growth indices reported showed an effective randomization for all heat treatments but the values of the growth indices varied enough so that it was difficult to determine if any one heat treatment was more effective than the others. Further analysis using inverse pole figures is being carried on. Dimensional changes, grain sizes, and growth indices observed in the samples which were oil quenched are all comparable to those observed in the nitrate quenched samples.

A marked columnar grain structure has been observed in some fuel element geometries after various heat treatments. In order to keep the grain structure as small and as random as possible in future heat treatments, information must be obtained concerning the variables contributing to the columnar grain structure. A variety of fuel geometries and as-extruded grain structures are being heat treated using a variety of heat treatments; cooling rates and thermal gradients across the samples are being measured using a Visicorder. The above variables will be correlated not only with columnar grain structure, but also with grain size, dimensional changes and preferred orientation.

Fabrication of Dual Enrichment Rods - R. G. Nelson

The quarterly report for April, May and June 1960⁽³⁾ describes the fabrication of simulated dual enriched rod type elements. Vacuum induction melted and heat treated uranium sleeve and core pieces were coextruded

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FIGURE 4.17

Rod from Cast and Heat Treated Components

FIGURE 4.18

100 X

FIGURE 4.19

Re [REDACTED] extruded Components Simulated Dual Enriched Rod Elements

FIGURE 4.20

100 X

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Two tubes have been discharged, one at 1200 MWD/T and the second at 2000 MWD/T exposure and post-irradiation examination has started. Examination of the discharged elements in the basin revealed no bumping or warping of a significant nature. The iron rails were black upon discharge, but started to corrode in the neutral basin water. The 1200 MWD/T pieces were covered with a light, sooty appearing deposit, and the 2000 MWD/T pieces were extremely clean but lacked the usual black luster of KER irradiated Zircaloy-2 material. No evidence of corrosion of the braze-weld closures was apparent on the 1200 MWD/T pieces (perhaps because of sooty deposit), but some white areas had developed on the 2000 MWD/T pieces.

The irradiation induced volume increases of the tube elements was determined in the K basin. The technique used required pre-irradiation weighing of the 7.5 kg elements, weighing the irradiated elements in the basin, and computation of the volume increase from the weight data. Volume increases for the 1200 and 2000 MWD/T pieces are listed in Table 4.3. Available data from radiometallurgical examination is also tabulated in Table 4.3.

TABLE 4.3

SWELLING AND DIMENSIONAL CHANGES OF KER SINGLE TUBES

Exposure	Position	Mean Fuel Temp. (°C)	Fuel Swelling Volume (Per Cent)	ΔLength (in.)	ΔOD (in.)	ΔWarp*** (in.)
1200 MWD/T*	Upstream	430	0.54	-0.054	+0.004	0.000
	↓	445	1.3	-0.075	+0.003	0.000
	to	455	1.3	-0.075	+0.003	+0.009
	Downstream	460	1.3	-0.076	+0.005	-0.002
2000 MWD/T**	Upstream	430	2.6	-0.027	+0.009	-0.001
	↓	445	3.0	-0.030	+0.010	0.000
	to	455	3.1	-0.034	+0.011	+0.004
	Downstream	460	3.0	-0.044	+0.013	+0.009

* 30 per cent of irradiation at low temperature.

** 10 per cent of irradiation at reduced temperature.

*** Single Throw warp = 1/2 double throw warp.

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Tubular Fuel Elements - G. T. Geering

Four KER tube-tube fuel elements were irradiated to 3200 MWD/T in a KER Loop. One of the elements with a uranium core failed and a description of the failure was given in the third Quarterly for 1960. (4) Two of the fuel elements had U-2 w/o Zr cores and the post-irradiation examination of the inner tube of one such element was reported in the fourth Quarterly for 1960. (5) The outer tube from the U-2 w/o Zr element was examined in a Radiometallurgy cell this Quarter. In contrast to the uranium outer tube examined previously, the U-2 w/o Zr outer tube has a smooth surface. A transverse section at the mid-length of the tube bears this out (Figure 4.21). Once again for a U-2 w/o Zr tube no cracks were found in the U-2 w/o Zr core of the transverse section. A summary of pre- and post-irradiation diameters is given in Table 4.5.

TABLE 4.5

EFFECT OF IRRADIATION ON KER U-2 w/o Zr TUBE RM 569 B-2

<u>Diameter</u>		<u>Upstream End</u>	<u>Midlength</u>	<u>Downstream End</u>	<u>Length</u>
0°	Pre OD	1.813	1.820	1.819	Pre 17.483
	Post OD	1.829	1.831	1.825	Post 17.5
90°	Pre OD	1.818	1.818	1.817	Pre 17.480
	Post OD	1.821	1.825	1.826	Post 17.5
0°	Pre ID	1.456	1.456	1.456	
	Post ID	-	1.451	-	
90°	Pre ID	1.456	1.456	1.456	
	Post ID	-	1.445	-	

The post irradiation inner diameters were determined by measuring the wall thicknesses of the transverse section (Figure 4.21) optically and subtracting the corresponding thickness values from the measured outer diameters. The wall thickness after irradiation varied from 0.185 to 0.197 inches over one transverse section. The pre-irradiation variation was from 0.180 to 0.182 inch for six measurements, two at each of three locations along the tube.

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Irradiation of Variable Heat Treatment Elements - R. S. Kemper and R. N. Johnson

Ten 15-inch elements of NPR inner tube stock, processed with variable beta heat treatment, were irradiated to an estimated average exposure of 1050 MWD/T. The outlet water temperature, at equilibrium, was 297-288 C. The ten elements will be examined in Radiometallurgy after an approximate one month cooling period.

Two elements of the same group are undergoing irradiation in the ETR 3X3 Loop in cycle 36.⁽⁶⁾ The elements are operating at outlet loop temperature of 230 C (443 F) and charge power of 108-110 kw, including basket power. The elements will be dimensionally characterized after the initial irradiation, using the measuring equipment currently being installed at NRTS.

Irradiation of Fuel Elements with Brazed Closures - J. C. Tverberg

Two irradiation experiments were performed during the last quarter on experimental fuel elements with brazed closures. Both experiments involved co-extruded fuel with the closure formed by brazing a Zircaloy end cap to jacket and uranium core using the 4 w/o Be, 12 w/o Fe, 84 w/o Zircaloy-2 brazing alloy.

Thermal and power generation conditions in the GEH-4 facility of the MTR provide an excellent condition for testing experimental fuel elements in low temperature water. By proper design of the core thickness, high thermal stresses can be induced in the fuel element between the relatively cool jacket and high temperature core. These stresses will in turn exert themselves on the closure. Experiment GEH-4-57, 58 was such a test. Fuel material was 1.430 OD by 0.520 ID and 8.3-inches long. The test consisted of two elements suspended vertically in a basket with element 57 as the bottom element and 58 as the top element. After accumulating 1050 MWD/T (0.11 a/o burnup or 6×10^{19} fissions/cm³) a cladding rupture occurred at the base of brazing heat affected zone, 1-1/2 inches from the end of element 57. Figure 4.22, taken in the MTR basin immediately after discharge, shows the nature of the rupture. Later examination in a hot cell indicated that rupture probably occurred from a pinhole in the cladding or from an extrusion fold. Two cracks were observed on the rupture blister. The first is a crescent shaped crack extending across the blister. This crack appears to match a slightly darkened area on a pre-irradiation autoradiograph. A second longitudinal crack probably occurred as a result of a split type failure and probably was responsible for scrambling the reactor as this is the longer crack. A detailed metallographic examination of the closure is planned as soon as the radioactivity had decayed to the point where the element can be safely handled. The irradiation data on this test from the previous Quarterly Report are again presented in Table 4.6 for reference.

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TABLE 4.6

IRRADIATION DATA FOR GEH-4-57, 58

Uranium Core Temperature	329 C (623 F)
Uranium Surface Temperature	184 C (363 F)
Maximum Jacket Temperature	103 C (217 F)
Inlet Water Temperature	38 C (100 F)
Maximum Heat Flux	5000,000 Btu/(hr)(ft sq)
Water Flow Rate	20 gpm
Water Velocity	34 ft/sec
Film Coefficient	6000 Btu/(hr)(°F)(ft sq)
Exposure at Time Rupture	1050 MWD/T
	0.11 a/o burnup
	6×10^{19} fissions/cu cm

A KER size tube-tube, high power, high temperature test was placed in the 6X9 Loop in the ETR. This test was designated as GEH-10-39, 40, 41, 42. A vertical thermal flux profile, shown in Figure 4.23, was obtained by inserting these elements in the ETR critical facility. The power generation was somewhat lower than the calculated value. A comparison of the calculated and actual conditions is presented in Table 4.7. After the outer tubes have accumulated an exposure of 1200 MWD/T (0.14 a/o burnup or 6.3×10^{19} fissions/cm³) and the inner tubes have accumulated 825 MWD/T (0.1 a/o burnup or 5×10^{19} fissions/cm³) the elements will be returned to Hanford for measurement and radiometallurgical examination.

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TABLE 4.7

IRRADIATION DATA FOR GEH-10-39, 40, 41, 42

Heat Flux (Maximum)	1,329,000 Btu/(hr)(ft sq)	1,100,000 Btu/(hr)(ft sq)
Heat Flux (Minimum)	830,000 Btu/(hr)(ft sq)	680,000 Btu/(hr sq)
Maximum Clad Temperature	416 C (781 F)	393 C (740 F)
Maximum Fuel Surface Temperature	563 C (1046 F)	538 C (1000 F)
Maximum Fuel Core Temperature	669 C (1240 F)	638 C (1190 F)
Outer Tube Burnup Rate	80.5 Mw/T	65 Mw/T
Inner Tube Burnup Rate	62.9 Mw/T	52 Mw/T
Inlet Water Temperature	235 C (455 F)	235 C (455 F)
Outlet Water Temperature	289 C (553 F)	268 C (513 F)
Maximum Coolant Velocity	33.4 ft/sec	
Minimum Coolant Velocity	21.4 ft/sec	
Loop Pressure	2000 psi	
Loop Flow Rate	150 gpm	156 gpm
Effective Flux Outer Tube	2.05×10^{14} n/cm ² -sec	1.67×10^{14} n/cm ² -sec
Effective Flux Inner Tube	1.46×10^{14} n/cm ² -sec	1.27×10^{14} n/cm ² -sec

Defect-Testing of Irradiated Coextruded Fuel Material - J. W. Goffard

A knowledge of the influence of irradiation upon the defect-test behavior of coextruded fuel material is being gained from IRP facility test (IRP= Irradiated Rupture Prototype, an out-of-reactor recirculating, high-temperature, high-pressure irradiated material and decontamination test loop operated by Coolant Systems Development). Coextruded Zircaloy-2 clad uranium fuel rods have been separated from irradiated cluster fuel elements, defected with 0.025 holes, and tested in the IRP. Examination of the ruptured test pieces have been made in the Radiometallurgy facility.

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TABLE 4.8

SUMMARY OF DEFECT-TESTS OF IRRADIATED COEXTRUDED FUEL RODS

<u>Test Conditions</u>	<u>Fuel Rod History</u>	<u>Fuel Loss After Defect Incubation</u>	<u>Remarks</u>
<u>Influence of Irradiation Exposure (at constant test conditions)</u>			
(1) 300 C isothermal	Unirradiated	12 g @ 60 min 40 g @ 120 min 65 g @ 180 min	Single blister developed; no failure of clad-bond zone.
(2) 300 C isothermal	1200 MWD/T in KER (~250 C)	14 g @ 40 min 42 g @ 30 min	Multiple blisters; failure in clad-bond zone.
(3)* 300 C isothermal	2250 MWD/T in KER (~250 C)	9 g @ 20 min 23 g @ 40 min	Multiple blisters; failure in clad-bond zone.
<u>Influence of Irradiation History (at constant test conditions)</u>			
(4) Normal shut-down after failure detection	2400 MWD/T in K through-hole (~35 C), plus 5 hours at 260 C in ETR	183 g after 38 min in cooling from 300 C to 220 C	Complete clad separation and multiple blisters 3-4 in. along rod; fuel fragmentation.
(5)** Normal shut-down after failure detection	2250 MWD/T in KER (~250 C)	15 g after 50 min in cooling from 300 to 200; 16 g after 79 min total shut-down time.	Clad-bond zone failure.
<u>Influence of Shut-down Rate (with constant fuel rod history)</u>			
(3)* 300 C isothermal	2250 MWD/T in KER (~250 C)	9 g @ 20 min 23 g @ 40 min	Multiple blisters; failure in clad-bond zone.
(5)** Normal shut-down after failure detection	2250 MWD/T in KER (~250 C)	15 g after 50 min in cooling from 300 to 200; 16 g after 79 min total shutdown time.	Clad-bond zone failure.
(6) Scram shut-down after failure detection	2250 MWD/T in KER (~250 C)	4 g after 20 min in cooling from 300 to 200 C; 6 g after 44 min total shutdown time.	Multiple blisters; limited clad-bond zone failure.

* Same test

** Same test

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2. Smith, E. A. A Detector for Flaws and Non-Bonds in Zircaloy Fuel Element Closures, HW-69653. February, 1961.
3. Cadwell, J. J. Quarterly Progress Report - Fuels Development Operation - April, May, June, 1960, HW-67904. July, 1960. (SECRET).
4. Cadwell, J. J. Quarterly Progress Report - Fuels Development Operation - July, August, September, 1960, HW-69095. October, 1960. (SECRET).
5. Albaugh, F. W. Reactor and Fuels Quarterly Report - October, November, December, 1960, HW-70355. January, 1961. (SECRET).

CERAMIC FUELS DEVELOPMENTHot Swaging of UO₂ - P. L. Farnsworth

A hot swaged Zircaloy-4* clad, four-rod cluster containing fused UO₂ is being irradiated in the MTR. Three of the fuel rods in the cluster were hot swaged, and the fourth was cold swaged. The hot swaged fuel rods contained -60 mesh fused UO₂, and were induction heated to approximately 850 C just before swaging. Bulk densities of approximately 93 per cent TD were obtained. The external oxide scale which formed on the fuel rods was removed by belt centerless grinding. Final wall thickness after grinding was 0.020-0.024 inch. The hot swaged fuel rods were cut to length and end caps were welded in place. The rods passed all quality control tests normally given PRTR fuel rods. (1) This irradiation test is designed to provide information about the irradiation behavior of hot swaged fuel and to indicate the extent to which any hydrogen present in the hot swaged cladding precipitates as embrittling hydride during irradiation.

Chemical analyses of several samples of hot swaged Zircaloy-4 cladding revealed that induction heated fuel rods picked up less hydrogen than did resistance heated fuel rods. The hydrogen concentration in the Zircaloy cladding heated by these two methods is shown in Figure 5.1. The reason for the higher hydrogen concentration on both surfaces of the resistance heated specimen is not known at present, but may be associated with the arcing which occurs between the contact brushes and the fuel rods during resistance heating. As shown in Figure 5.2, however, hydride cannot be detected metallographically in samples of material heated by either method.

A Zircaloy-4 tube was loaded in a helium atmosphere with outgassed -60 mesh fused UO₂ and another Zircaloy-4 tube was loaded in an air atmosphere with the same material. End caps were welded in place after both tubes had been evacuated and backfilled with helium. The tubes were induction heated to approximately 850 C and hot swaged. Chemical analyses of turnings from each tube revealed no significant difference in hydrogen pick-up. This indicates that the outgassed UO₂ does not rapidly sorb water vapor from the air and that the UO₂ can be safely loaded in the tubes in an air atmosphere if the loaded tubes are immediately evacuated and backfilled with helium. As expected, no hydride was detected metallographically.

Metallographic examination of a section of an autoclaved (315 C, 1500 psi, 24 hours) hot swaged Zircaloy-4 clad fuel rod showed no hydride platelets present, indicating that the hydrogen in the cladding was not precipitated by the autoclave treatment.

* Zircaloy-4 as used in Section 5 refers to early formulation, nickel-free Zircaloy-2.

A. Resistance Heated

B. Induction Heated

FIGURE 5.2

Resistance Heated and Induction Heated Zircaloy-4 100X

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PRTR Thermocoupled Fuel Rods - L. E. Mills, L. R. Vancott

The Mark I PRTR fuel element design was modified to incorporate six thermocouples located within the UO_2 core to indicate fuel temperatures during irradiation. The chromel-alumel thermocouple wires were insulated by an aluminum oxide core sheathed with 0.010 inch wall by 0.062 inch OD, 304 L stainless steel tubing. The end of the thermocouple was positioned in the center of the fuel before swaging the Zircaloy clad fuel rod. After swaging, the end of the thermocouple was accurately located within the UO_2 fuel with a 2 Mev radiograph. The fuel rod was cut to length and machined to accept special end closure caps. The stainless steel clad thermocouple extended through the center of the Zircaloy end cap at the top of the fuel element. A stainless steel extension was diffusion bonded to the Zircaloy end cap. The stainless steel clad thermocouple was then brazed to the stainless steel section of the end cap with a high temperature brazing alloy (Cr, 13.0 per cent; P, 10.0 per cent; C, 0.15 per cent maximum; Ni, remainder).

The six rods were assembled in a nineteen-rod cluster with three thermocoupled rods placed in the inner circle of rods and three thermocoupled rods placed in the outer circle of rods, as illustrated in Figures 5.3 and 5.4. The location of the thermocouple within each rod is shown in Table 5.1.

TABLE 5.1PRTR FUEL ELEMENT 1072-TC-1

Thermocouple Location Within Fuel Core

<u>Rod No.</u>	<u>Vertical Location</u>	<u>Horizontal Location</u>
2182	9-7/8 inches below center of fuel	1/16 inch from $\mathbb{C}$ of rod
2183	12-3/4 inches below center of fuel	3/32 inch from $\mathbb{C}$ of rod
2184	9 inches below center of fuel	1/64 inch from $\mathbb{C}$ of rod
2186	15-1/2 inches below center of fuel	1/64 inch from $\mathbb{C}$ of rod
2187	9-1/2 inches below center of fuel	3/32 inch from $\mathbb{C}$ of rod
2188	12-3/8 inches below center of fuel	7/64 inch from $\mathbb{C}$ of rod

FIGURE 5.4

Thermocoupled Fuel Element Cross-Section, PRTR Element
Number 1072-TC-1

TABLE 5.2

EFFECT OF PARTICLE SIZE DISTRIBUTION ON COMPACTED DENSITY
OF FUSED UO₂

Mesh Size (Tyler Series)	Mixture Composition, Weight Per Cent			
	IA, IB	IIA, IIB	IIIA, IIIB	IVA, IVB
-6 +8	18	18	---	---
-8 +10	18	37	---	---
-6 +10	---	---	55	48
-10 +14	18	6	---	---
-14 +20	6	6.5	---	4
-10 +20	---	---	12.5	---
-28 +35	5	---	---	---
-35 +48	5	4	---	---
-48 +65	5	8.5	---	---
-28 +65	---	---	---	24
-35 +65	---	---	12.5	---
-200 +270	9	---	---	---
-270 +325	8	---	---	---
-200	---	20	20	---
-325	8	---	---	24

Mixture	Compacted Bulk Density, Per Cent TD							
	IA	IB	IIA	IIB	IIIA	IIIB	IVA	IVB
8 min. vibration	83	83	82	82	84	84	84	84
8 min. vibration plus rapid cycling	88	88	89	89	89	90	88	89

Gamma absorptometer measurements of the rods containing the compacted UO₂ show less density increase in the bottom 12-18 inches than in the remainder of the rod. This effect does not occur if the fuel material is compacted using a rapid frequency sweep (5 per minute) and increasing acceleration during loading and compaction. Thus, the highest density measured (IIIB, 90 per cent TD) is not necessarily the highest density to which the mixture can be compacted, and mixtures of fused UO₂ having the same particle size distribution were in fact compacted to uniform densities of 92-93 per cent TD by rapid frequency sweeping.

TABLE 5.3

HIGH ENERGY IMPACT-FORMED UO₂ FOR
NESTED TUBULAR FUEL ELEMENT IRRADIATION

	Fuel Element Component		
	Rod	Inner Tube	Outer Tube
Particle Density, by mercury displacement			
g/cc	10.71	10.32	10.46
% TD	97.6	94.1	95.4
O/U ratio (coulometric analysis)	2.009	2.006	2.005
Surface Area, m ² /g	0.199	0.014	0.044
Gas: volume extracted at 1000 C, cc/g	0.117	0.026	0.043
ppm by weight H ₂	5	0.1	0.53
ppm by weight N ₂	5	10	0.1
ppm by weight O ₂	1.3	2.5	3
ppm by weight CO	13	9	26
Tyler Sieve Analysis, w/o +6 mesh	0.40	8.76	9.09
-6 +8	13.14	9.94	12.57
-8 +10	18.29	14.37	17.32
-10 +20	21.03	31.51	17.14
-20 +35	5.96	3.06	2.46
-35 +65	9.36	13.51	16.11
-65 +100	2.55	0.73	1.74
-100 +200	1.93	0.95	1.32
-200	22.54	16.67	22.25
Spectrographic analysis, ppm Al	200	200	100
B	0.1	0.1	0.1
Cr	50	2	2
Cu	100	10	5
Fe	480	115	102
Mg	2	2	1
Mn	50	10	10
Mo	5	5	5
Ni	100	5	5
Si	39	18	12

Electron Microscopy - J. L. Daniel, S. J. Walter, J. O. McPartland

Detailed examination and characterization of fused UO_2 single crystals were initiated. Both polished and natural cleavage surfaces were studied using transmission replicas and direct (reflection) techniques. Polished and etched specimens have shown etch pits and inclusions ranging from a fraction of a micron to 10 microns. These imperfections appeared to be grouped at "preferred locations" throughout the crystals, the size and frequency of groups differing in each crystal (Figure 5.6). The inclusions have not yet been identified.

Cleavage surfaces of fused UO_2 single crystals are particularly appropriate for reflection electron microscopy; the surfaces are naturally glass-smooth except for the surface structure of interest. Reflection micrographs of several crystal surfaces were made, both at room temperature and at elevated temperature. Only minor changes, primarily vaporization of some of the surface debris originally present, occurred in a UO_2 crystal heated between 1000 and 1150 C for one hour. Examination by optical microscopy showed evidence of fusion and other heat effects along edges of the specimen, suggesting that the temperature in the area on which the electron beam impinged was considerably above that indicated by the thermocouple at the sample surface adjacent to the heater.

An experimental furnace was designed for heating transmission samples to 1800-2000 C in the electron microscope. Lucalox supporting members and a tungsten heating grid will be used. Sample interchange will be possible through the existing sample port of the microscope. Temperature will be determined by calibration of the direct current supply or by optical pyrometry through a viewing port.

Thermal Conductivity of UO_2 - J. L. Daniel, S. J. Walter

Measurements were completed at Battelle on an irradiated and a non-irradiated UO_2 specimen (Figure 5.7). The irradiated UO_2 (3.48×10^{19} nvt) showed a lower thermal conductivity and a more prominent minimum at about 175 C than previously measured for a lower exposure specimen.

Thermal conductivity of a cold-pressed and sintered specimen (3-inch diameter by 7/8-inch thick) was measured to 1400 C. Data define a curve of continuously decreasing "k" with increasing temperature. The curve is slightly above the one previously obtained for an extruded, isostatically pressed and sintered UO_2 rod. A visible crack occurred in the disc at about 1200 C; measurements thereafter were uniformly about 10 per cent lower than before.

Additional specimens for thermal conductivity measurements to 1400 C include a single crystal of fused UO_2 (0.3 x 0.4 x 1.8 inches) and a 3-inch diameter disc of fused UO_2 grain vibrationally compacted to 86 per cent TD.

FIGURE 5.7
Thermal Conductivity of Irradiated and Nonirradiated UO₂
(Corrected to Zero Porosity)

point of all the other claddings investigated, but only in the case of Fansteel 80 was it sufficiently extensive to prevent removal of the UO_2 specimen from the cladding after heating. The Fansteel 80 strip broke after 3 minutes 45 seconds at 2290 C, and the UO_2 appeared to have reacted almost completely

Tubular UO_2 Fuel Element Irradiation (HAPO-6) - M. K. Millhollen, G. R. Horn

A vibrationally compacted UO_2 tubular fuel element was irradiated successfully in a high temperature, high pressure Hanford reactor test facility. The 18-inch long Zircaloy-2 clad fuel element (1.91-inch OD x 0.66-inch ID x 0.06-inch clad) contained natural, fused UO_2 vibrated to a bulk density of 98 per cent of theoretical. The fuel length was 17.44 inches, and the gap between fuel and top end cap was 1/16 inch.

This fuel element (Figure 5.8) was spaced in the process tube by Zircaloy-2 bridge spacers spot welded to the cladding.

Nested Tubular Fuel Element Irradiation (GEH-4-22) - M. K. Millhollen, R. C. Smith, G. R. Horn

A 44-inch long, prototypic Mark IIC nested tubular fuel element was fabricated by vibrational compaction for irradiation testing in the ETR. The element was clad in 0.060-inch wall Zircaloy-2 cladding having integrally formed spacer ribs. The depleted UO_2 fuel core was prepared by the previously described (page 5.10) high energy impact process, followed by a low temperature reduction step.

Each of the three fuel sections was fabricated and inspected separately before assembly into the fuel element. Filling and compaction of the outer fuel tube, and the fuel element components before assembly are shown in Figure 5.9. End caps were welded to the tubular sections by a seam weld and by a fusion weld. All sections, Figure 5.10, of the element passed a leak test, a weld radiograph test, and autoclaving for 24 hours in 400 C, 1500 psi steam.

Thermocouples were attached to the three ribs of the inner tube with 0.020-inch diameter stainless steel wire. The three units were assembled and welded to the hanger fittings (Figure 5.11). Following irradiation, the fuel element will be returned to Hanford for examination.

FIGURE 5.9

Fabrication of Prototypic Nested Tubular Fuel Element

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FIGURE 5.11
Assembled Prototypic Mark IIC Nested Tubular Fuel Element

Reduced 4.8 X in Printing

FIGURE 5.12

Irradiated Partially Decontaminated, Recycled UO_2

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END