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ATR FUEL MATERIALS DEVELOPMENT IRRADIATION RESULTS -- PART II

by

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ABSTRACT

Irradiation experiments upon fuel materials selected for use in the Advanced Test Reactor and the High Flux Isotope Reactor and possible substitutes were executed at conditions prototypical of full-power operation in a loop facility in the Engineering Test Reactor. The fuel materials behaved satisfactorily with respect to fuel dimensional stability during the irradiations. The materials tested included

- (1) 41 weight percent U_3O_8 with 0.19 weight percent B_4C
- (2) 51 weight percent UAl_3 with 0.19 weight percent B_4C
- (3) 55 weight percent UAl_3 with 0.19 weight percent B_4C
- (4) 56 weight percent Nb-coated UO_2-ZrO_2 with 0.16 weight percent B_4C
- (5) 41 weight percent U_3O_8 .

The matrix and cladding materials for all compositions were X8001 aluminum and 6061-0 aluminum, respectively. The measured in-pile corrosion rate of 6061-0 cladding was found to agree reasonably well with the measurements made ex-reactor by J. C. Griess at ORNL[1].

Post-irradiation anneal experiments on plates containing the nominal ATR fuel and poison loadings showed the threshold temperature for breakaway swelling to be 750°F for the U_3O_8 fueled plates and 800°F for UAl_3 plates which had a fission density of 15×10^{20} f/cc. The breakaway swelling temperature for a Nb-coated UO_2-ZrO_2 plate with the same U-235 and B-10 content was 500°F with a fission density of 11×10^{20} .

SUMMARY

The work described in this report is a continuation of an irradiation program conducted upon fuel materials of interest to high-performance test reactors such as the Advanced Test Reactor (ATR) and the High Flux Isotope Reactor (HFIR). A series of proof-tests conducted previously showed that blisters formed on sample fuel plates at fission densities above about 8×10^{20} fissions per cubic centimeter (f/cc) while operating at temperatures which were not known with satisfactory precision. A loop facility (known as PAED-G-12 ETR Water Loop) was designed and constructed in which more carefully controlled irradiation experiments were executed. The operating conditions that could be achieved corresponded to the locations in the reactors which were operating at the maximum nominal power density (ie, not including off-normal contributions known as hot spot-hot channel factors).

The fuel materials tested, the maximum nominal cladding temperature and surface heat flux, and the average fission densities obtained are as shown in the following tabulation:

Fuel Material	Maximum Nominal Cladding Temperature (°F)	Maximum Nominal Surface Heat Flux 10^6 Btu/hr-ft ²	Fission Density 10^{20} f/cc
41 wt% U ₃ O ₈ in X8001 plus 0.19 wt% B ₄ C (ATR Reference)	360 to 470	1.9 to 2.0	7.1 to 16
41 wt% U ₃ O ₈ in X8001 (HFIR Reference)	310 to 365	1.3 to 1.4	4.6 to 11
51 wt% UAl ₃ plus 0.19 wt% B ₄ C in X8001	385 to 470	1.8 to 2.0	7.1 to 16
55 wt% UAl ₃ plus 0.19 wt% B ₄ C in X8001	310 to 330	1.5 to 1.7	6.4 to 14
56 wt% UO ₂ -ZrO ₂ Spherodized, Nb-Coated, plus 0.16 wt% B ₄ C in X8001	390 to 515	1.8 to 2.2	8.1 to 17

All the fuel samples were clad with 6061-0 aluminum.

No evidence of blister formation or other deleterious effects attributable to fuel material stability was observed during the irradiations except that one of the UO₂ fueled plates became severely warped; the cause for this behavior is believed to be the poor distribution of fuel within this particular plate.

Measurements of the cladding temperature of instrumented plates during the course of the irradiations showed that the corrosion of the 6061-0 aluminum agreed reasonably well with a correlation developed from ex-reactor measurements[1]. Post-irradiation measurements of the alumina thickness tended to corroborate the in-pile temperature data. Hence, the ex-reactor correlation is recommended for use in predicting ATR fuel plate corrosion performance.

Post-irradiation metallurgical studies provided the following conclusions:

- (1) The reference fuel (U_3O_8) would be satisfactory for use under the currently anticipated ATR and HFIR operating conditions which are similar to those obtained during the test.
- (2) The use of powder UAl_3 as a fuel material in these reactors would offer a greater margin of safety
- (3) The use of Nb-coated UO_2 particles is not recommended for use in these reactors.

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ATR FUEL MATERIALS DEVELOPMENT IRRADIATION RESULTS -- PART II

I. INTRODUCTION

During the design of the Advanced Test Reactor (ATR), it was recognized that the fuel material to be used in the core fuel elements could not be the uranium-aluminum alloy successfully employed in the Engineering Test Reactor since the fuel loading required was greater than could be successfully produced commercially and since adding the metallic boron burnable poison had been found to be difficult and generally marginal in ETR fuel elements. Use of powder metallurgical techniques appeared requisite to circumvent these problems. At that time (1962), limited irradiation experiments upon uranium oxide-aluminum cermet had been performed [up to 7×10^{20} fissions per cubic centimeter (f/cc)] at relatively low temperatures (about 100°C) with no evidence of fuel stability problems. It was clear, however, that the service requirements in ATR would surpass the conditions of these preliminary experiments and the cermet fuel behavior for these more stringent conditions might not be as favorable. Accordingly, an experiment was devised to essentially proof-test the selected fuel material (41 weight percent U_3O_8 in X8001 aluminum with 0.19 weight percent B_4C added and clad with 6061 aluminum alloy) together with possible substitutes.

The most expedient means by which a proof-test of the reference fuel could be executed was to insert the samples in a holder in the ETR core in a high flux position and to cool the samples with ETR primary coolant since a recirculatory loop was not available and since installation of a loop was considered unnecessary. To obtain a fuel matrix temperature corresponding to that expected during reference full-power operation of ATR, it was necessary to apply a thermal barrier to the surface of the sample fuel plates; this was accomplished by anodizing. Subsequent to the irradiation of the reference fuel and the possible substitutes (35 weight percent UO_2 or 50 weight percent UAl_3 -- otherwise the same as the reference or clad with X8001), it was found that blistering of the U_3O_8 - and UO_2 -bearing plates had occurred between about 7×10^{20} and about 12×10^{20} f/cc. Consideration of the behavior of the thermal barrier showed that neither the corrosion rate of the cladding, adding to the thermal barrier, nor the thermal conductivity of the barrier when exposed to a radiation environment were understood. Consequently, the temperatures achieved by the fuel core during the irradiation were in doubt. Reference 7 describes the results of this experiment.

To allow irradiation experiments to proceed safely with nominal beginning-of-core-life, surface temperatures of 350°F, it was apparent that an increase in the coolant pressure in comparison to that available in the ETR primary system was needed to provide an adequate margin to thermal burnout. A loop facility of some kind was indicated. In the interests of economy and of reproducing the water chemistry maintained in test reactor systems and anticipated for ATR operations, it was decided that pressurization of a side-stream of ETR primary coolant offered the most desirable scheme. The loop facility which was designed, constructed, and operated for this experiment is described in this report.

The purposes of the test described in this report were essentially twofold. The foremost objective was the determination of the stability of the reference ATR fuel material and of several possible substitutes. The fuel materials tested included

- (1) 41 weight percent U_3O_8 with 0.19 weight percent B_4C added in X8001 aluminum (the ATR reference fuel material)
- (2) 50 and 55 weight percent UAl_3 with 0.19 weight percent B_4C added in X8001
- (3) Spherodized and niobium-coated 56 weight percent UO_2-ZrO_2 with 0.16 weight percent B_4C added in X8001
- (4) The reference fuel omitting the B_4C (HFIR reference fuel material).

All samples were clad with 6061-0 aluminum since its corrosion rate in ex-reactor experiments was found to be essentially the same as X8001[1] and, since, at the expected temperatures, 6061-0 has better mechanical properties[2].

The secondary objective was to confirm the corrosion rate of 6061-0 aluminum as measured in ex-reactor experiments[1] since there was sketchy evidence that the in-reactor corrosion rates might be less than those measured ex-reactor. Corrosion of the cladding, per se, appears important only because of the insulating character of the adherent corrosion products thus increasing the fuel and cladding temperatures during operation; the fuel plate cladding thickness is about an order of magnitude larger than the anticipated corrosion; hence, the fuel core alloy will not be exposed to the coolant.

II. EXPERIMENT DESIGN

1. PAED-G-12 ETR WATER LOOP

1.1 General

To achieve conditions of fission rate and coolant velocity in the fueled channels and, therefore, fuel plate surface temperature prototypical of the ATR, it was necessary to employ the flux trapping principle and to select ETR fuel elements and preferentially place them in the ETR core. The experimental facility occupies a 3 by 3 corner (ETR core position G-12) of an ETR 6 by 6 through facility (so-called G-13 facility), and significant changes in the incident neutron flux may be made by placing standard fuel elements in the other 3 by 3 positions of the facility.

The necessary loop water conditions are provided by taking a side stream of ETR primary coolant from above the ETR core, increasing the coolant pressure and grossly controlling the flow rate in the main loop pumps, passing the loop coolant through the in-pile experimental section, and returning the loop effluent to the ETR reactor primary discharge line. The design conditions of the loop equipment are as follows:

Temperature -- 300°F

Flow Rate -- 200 gpm

Pressure -- 900 psig

The principal materials of construction are as follows:

Piping and fittings -- Type 347 stainless steel

Valve bodies -- Type 316 stainless steel

Pump casings -- Carbon steel

Pump impellers -- Type 347 stainless steel

Delay tanks -- Type 347 stainless steel

In-pile tube -- Zircaloy-2.

The codes used in the design were ASA B31.1 and ASME Section VIII for Lethal Substances. All welds one inch o.d. and larger were inspected in accordance with the local standards for in-pile experimental equipment; these require radiographs of the root and final passes of each of the welds.

Since Section VIII of the ASME Code does not include Zircaloy-2, the intent of the code was employed in that one-quarter of the ultimate stress or one-half of the yield stress at 0.2 percent offset (whichever is smaller) was used as the maximum allowable stress. The in-pile tube was not pre-oxidized since the service temperature was considered insufficient to cause significant corrosion.

ASA Code Case N-10 for the valve bodies was modified in that a minimum number of radiographs and dye penetrant examinations to the maximum extent possible were required to give reasonable assurance that in-service failures would not occur.

Finally, the completed loop was hydrostatically tested at 900 psig since original operation was executed at less than 600 psig.

1.2 Detailed Description

1.21 Coolant System. Loop coolant is supplied through a two-inch pipe inserted vertically into the ETR reactor vessel and which terminates about six feet above the ETR core; a four-inch-diameter cylindrical perforated strainer is attached to the suction to prevent passage into the loop of foreign matter greater in size than 1/8 inch diameter. The supply coolant flows by gravity or by the ETR operating inlet pressure (200 psig) through a three-inch pipe (outboard of the reactor vessel flange) through a gate valve to the suction side of either of two vertical, four-stage centrifugal pumps. The coolant pressure is increased by 700 ft (at 150°F) in either pump. A check valve and a globe valve, used to regulate the pressure in the in-pile tube, reside in the three-inch line between the pump discharge and the in-pile tube. A crossover line, containing a check valve between the pump's suction and main discharge line, is provided to permit flow through the in-pile tube using the ETR core differential pressure as the driving force.

The coolant flows downward through the experimental section by means of a two-inch-inlet and -outlet stainless steel pipe and the Zircaloy-2 in-pile tube; outboard of the reactor vessel, the discharge piping is three inch. The coolant flow rate is measured by a sharp-edged orifice and is regulated by a flow control valve; both are mounted in the discharge line between the reactor vessel and the first of two 200-gallon delay tanks. During normal operation, the coolant flows through the second delay tank, two solenoid-operated gate valves in series, a manually operated gate valve, a check valve, another manually operated gate valve (located in the ETR primary coolant pipe tunnel), and into the ETR primary coolant reactor discharge line. Figure 1 depicts schematically the equipment layout.

In the event a major fission break is detected by either the delayed neutron monitor or the direct gamma monitor, the loop effluent is diverted to the hot waste storage tank by closing the solenoid-operated gate valves in the return line and simultaneously opening two solenoid-operated gate valves mounted in parallel in a line taking off from the line between the delay tanks. The characteristics of the trims of these four valves and solenoid actions regulate the flow such that the series pair do not close before the parallel pair open, thus assuring that flow through the experimental assembly is not disrupted. Either gross-fission-break monitor actuates a time-delay relay which after a preset increment causes the flow control valve to close to a preset stop, thereby minimizing the volume of hot effluent discharged to the hot waste storage tank farm.

Minor fission breaks are identified by means of an iodine fission-break monitor which may monitor loop effluent or the ETR primary coolant prior to its admission into the experimental section.

A side stream from the pump discharge flows through a pH cell and provides the information from which manual control of the rate of acid injection into the pump suction is obtained.

Loop coolant samples may be obtained from either the loop influent or effluent.

1.22 In-pile Equipment. The design criteria for the in-pile equipment include

- (1) A material in the core region which could withstand the pressure and thermal stresses expected during operation and yet have a minimum neutron absorption cross section
- (2) Provision for remote, wet insertion of irradiated and instrumented samples.

Fully annealed Zircaloy-2 was specified as the material for the in-pile tube since it has a small neutron absorption cross section combined with acceptable mechanical properties although its lifetime in operation appeared to be limited by hydriding from proton recoil. The combined pressure and thermal stresses (from gamma heating) were calculated to exceed substantially any of the commercially available aluminum alloys thus eliminating them from further consideration. The neutron attenuation expected from practical thicknesses of Type 347 stainless steel caused this material to be discarded. The selection of Zircaloy-2 did impose a problem of attaching it to the stainless steel loop piping. This was accomplished by means of mechanical joints located above and below the core. These joints are essentially an ordinary pipe union with a metal "O-ring" seal added to give further assurance that the joints will not leak. Figure 2 is a drawing of the in-pile equipment. In the core region, the Zircaloy-2 tube

Fig. 2 PAED-59 core section assembly.

is 2.4 inches o.d. with a 0.15-inch wall thickness; immediately below the core, the tube is tapered to an outside diameter of 2.25 inches to obtain an annulus formed by the tube and the ETR grid plate of 0.125 inch, thus assuring adequate flow external to the tube. The tube proper was extruded; whereas, the mechanical joints were machined from billets and welded to the tube.

Ex-reactor tests upon a prototype stainless-to-Zircaloy-2 mechanical joint were executed prior to their in-pile use. The prototype joint was subjected to five hundred pressure cycles from 0 to 600 psig each; five hundred temperature cycles from 80 to 270°F each; and two million mechanical deflection cycles, each

cycle being comprised of applying, removing, and reversing a moment of 500 inch pounds. Subsequent to the cyclic exposures, the joint was hydrostatically pressure tested at 1000 psig with no evidence of leakage.

The second criterion was achieved by extending the in-pile tube vertically with 2-1/2-inch pipe to a closure plug and admitting the coolant to the in-pile tube through a 2-inch line joining the vertical extension at a 45 deg angle. The top closure plug is 4 inches o.d. and 6 inches long and is constructed to permit an Amphenol connection of the sheathed thermocouples as well as providing a double "O-ring" seal for the loop coolant (see Figure 3). Thermocouple extension lead wires are connected to a mating Amphenol and are routed through a nitrogen-pressurized flexible tube to a junction box external to the reactor vessel.

Fig. 3 Upper closure plug assembly.

The Zircaloy-2 in-pile tube is mechanically joined to a length of 2-inch pipe below the grid plate. This pipe contains a dogleg to suit the reactor vessel bottom head penetration and is precision machined and chrome plated in the region of the bottom head penetration to assure that an adequate seal is obtained. The bottom head closure plug is shown in Figure 4; it is a conventional design.

An aluminum core filler piece (see Figure 2) surrounds the in-pile tube in the core region thereby establishing the rectangular geometry required in the core region for reasons of structural performance of the ETR fuel elements. In addition, the core filler piece establishes an annulus 0.125 inch thick outside the in-pile tube. ETR primary coolant flow through this annulus using the core pressure differential as the driving force removes the gamma heat generated in the in-pile tube.

Fig. 4 Bottom head penetration.

1.23 Sample Support Assembly. Two not necessarily compatible requirements imposed by the irradiation program resulted in the design of the sample support assembly shown in Figure 2. The two requirements were

- (1) The temperature of several of the fuel plates must be monitored
- (2) In-canal inspection and fuel plate exchange must be allowed.

The support assembly design resulting consists of a U-shaped aluminum piece about 37 inches long in which instrumented plates 0.150 inch thick are mounted and six U-shaped pieces (each about 6 inches long) in each of which three sample fuel plates (each 0.050 inch thick) are mounted. The inner sides of each holder are notched to accomodate fuel plates 1.25 inches wide and which thereby establish five coolant channels, each of which are 1.10 inches wide and 0.100 inch thick. The notches are 0.005 inch thicker than the fuel plates to allow underwater removal and inspection of the latter. The edges of the holders are V-shaped, male and female, to obtain mechanical attachment of the short holders to the long holder.

There is a cylindrical pedestal welded to the bottom of the instrumented plate holder to form the transition from the rectangular sample assembly to the inside diameter of the in-pile tube. The pedestal has eight 3/32-inch holes drilled through it to provide regulated flow in the space between the holder and the in-pile tube, and a ring is welded on the bottom to center the assembly in the support tube. This design serves two purposes:

- (1) To minimize the flow outside the fueled channels which, if significant, would confound the heat balance comparison obtained in operation with that calculated from ETRC data
- (2) To maintain a pressure differential on the sample plate holder assembly from the outside-in thus assuring inward leakage.

The forces imposed upon the sample fuel plate holder assembly developed by the pressure drop across it are transmitted to the in-pile tube by means of a sleeve (a length of Schedule 5, Type 347 stainless steel pipe). This sleeve mates with the ring on the bottom of the circular pedestal and extends downward to a support ring in the lower stainless steel to Zircaloy mechanical joint.

1.3 Instrumentation

1.31 General. Three general purposes are required of the loop instrumentation:

- (1) To provide information from which the actual performance of the experiments can be controlled and evaluated
- (2) To sense the loop operating conditions and annunciate the occurrence of an off-normal condition
- (3) To cause an ETR power reduction in the unlikely event that the experiment or the reactor may be compromised.

The relatively high operating heat fluxes anticipated (up to $2\frac{1}{2} \times 10^6$ Btu/hr-ft²) together with the low heat capacity of the sample fuel plates resulted in relatively short adiabatic rise times of the plates and caused the specification of commercially available instrumentation with superior response time characteristics. Even so, it was not possible to obtain flow measuring instrumentation with adequate response time to minimize the effects of a loss-of-coolant-flow event, and direct-acting relays were installed in the loop pump electrical supplies to provide adequate protection.

1.32 Primary Coolant Instrumentation. Figure 1 shows the parameters which are monitored and annunciated and which cause reactor shutdown; also shown is their general location.

During installation of the unbonded strain gage transducers used to monitor system pressure and pressure differentials, it was found that operation of a dc welding machine in close proximity to the transducers damaged the latter to the extent that they had to be returned to the supplier for complete rework. Subsequent to this exposure, the transducers behaved as if they had been subjected to extreme overpressure -- roughly five times nominal.

The readout instrumentation and annunciators are mounted upon a central control panel as indicated in Figure 1. Each parameter which can initiate a reactor power reduction is recorded continuously and has a test block between the initiating contact and the loop reactor power reduction relays. Removal of a given test block permits maintenance upon that particular system. The system employs the auctioneering philosophy in conjunction with redundancy of each of those parameters which may cause a reactor shutdown; eg, the in-pile differential pressure provides a separate measurement of the coolant flow rate through the experimental assembly in addition to the direct measurement of the flow rate with a sharp-edged orifice. Removal of the test blocks of two related, redundant parameters causes a reactor power reduction; this feature is included to assure that operations do not proceed with inadequate protection.

1.4 Electrical

Both diesel and commercial power supplies the loop equipment. Under normal operational circumstances, the control panel and associated sensors, transmitters, power supplies, etc, are supplied from the commercial power supply; the operating loop pump is supplied from the diesel bus. Upon failure of either, a reactor scram is initiated, and the supply is switched automatically to the other.

The operating loop pump is supplied from the diesel bus to assure that upon commercial power failure and subsequent coastdown of the main ETR primary coolant pumps, the loop pressure and flow do not decay simultaneously. Thus, an unnecessary approach to thermal burnout is avoided.

1.5 Instrumented Fuel Plate

Thermocouples are installed in specially constructed sample fuel plates to measure the plate midplane metal temperature.

These plates consist of two 10-mil-thick fuel "meat" sections clad on both sides with 15 mils of cladding material. These two 40-mil plates are then assembled with a 70-mil aluminum filler piece in the center, thus making a plate with an overall thickness of 150 mils and with a 100-mil-thick aluminum center core into which the thermocouple is inserted.

A specially designed drill jig that makes use of a liner bushing and slip-removable drill bushings is used to drill the hole to a high degree of accuracy (as to both location and hole size) for the thermocouple on the plate midplane.

The thermocouples used are stainless steel clad and are fabricated with a nominal o.d. of 0.0625 inch. The hot junction is integral (grounded), and the o.d. at the hot-junction end is dressed down to conform to size specification.

Prior to insertion, the end of the thermocouple, for one inch of length, is "sized" in a split-block sizing die to an o.d. of 0.0624/0.0625 inch.

The plate is sequentially drilled, making use of the slip-removable drill bushings, with a No. 54 (0.0550 inch), No. 53 (0.0595 inch) and 1/16 inch (0.0625 inch nominal) to the required depth. However, the final size (1/16 inch) drill is slightly reduced in o.d. by dressing down the flute edges with a piece of fine aluminum oxide cloth held in thumb and forefinger while the drill is turning in the drill motor. All drilling is done with a portable 1/4-inch drill motor and is done in an inverted position so that the chips may fall out when the drill is dragged from the hole for cleaning of the flutes. Final size of the dressed-down drill is 0.0623/0.0622 inch. The first No. 54 hole is drilled in small depth increments of about 3/32 inch (cleaning the flutes and applying a light coat of lubricant to the drill each time). Subsequent drilling to No. 53 and 1/16 inch is accomplished in about 3/16-inch-depth increments, cleaning and lubricating the drill each time as before. The bottom of the drilled hole is squared by cleaning it out by hand with a square-ended drill in a pin vise to achieve a square-bottomed hole for minimum hot-junction contact resistance.

The thermocouple is driven into the hole with a standard aircraft riveting gun by employing a specially designed split clamp bar which grips the thermocouple and fits over the flush rivet set in the rivet gun. The thermocouple is lightly lubricated prior to insertion. About 90-psig air supplies the gun, but this

is additionally throttled with the needle valve on the gun until the "feel" is about right for clean insertion to full depth.

Careful lengthwise placement of the split clamp bar for driving the thermocouple and careful use of the gun will enable the operator to obtain a slight mushrooming or swelling of the thermocouple over a distance of about 1/8 inch after the thermocouple is bottomed (just outside the hole or plate edge) thus effecting a final tight seal in the plate.

2. FUEL SAMPLES TESTED

2.1 Sample Preparation

The sample fuel plate compositions used for the experiment are listed in Table I. As can be seen in the table, three of the five compositions were made

TABLE I
ATR FUEL IRRADIATION PROGRAM -- SAMPLE COMPOSITIONS^[a]

Composition Number	Fuel Material	Fabricator	Reduction Ratio	Loading			
				U-235		B ₄ C	
				g/Plate	g/in. ² Core	g/Plate	g/in. ² Core
1	UAl ₃ Powder	PPCo	8:1	2.2	0.40	0.013	0.0024
2	U ₃ O ₈	PPCo	8:1	2.2	0.40	0.013	0.0024
3	U ₃ O ₈	ORNL	9:1	2.2	0.40	---	---
4	Spherical UO ₂ + ZrO ₂ coated with Cb	ORNL	9:1	2.2	0.40	0.013	0.0024
5	UAl ₃ Powder	PPCo	8:1	2.4	0.44	0.013	0.0024

[a] All plates were clad with 6061-0 aluminum alloy. X8001 aluminum alloy powder was used as the core matrix material.

by the Metallurgy Laboratory Section of Phillips Petroleum Company, and two of the compositions were made by the Metals and Ceramics Division of ORNL. The reduction ratio used in the fabrication of the samples was chosen so as to be representative of that used in the fabrication of the full-size ATR and HFIR fuel plates. The fuel and burnable poison loadings were also chosen to represent those of interest for these reactors.

2.11 Fuel Materials Used. In Table II, the chemical analysis of the fuel materials used in the samples is shown. The U₃O₈ was supplied by the Y-12 Plant at Oak Ridge, Tennessee. It was identical to the oxide used in the first power element of the HFIR^[3]. It was made to conform to the following specifications:

TABLE II

ATR FUEL IRRADIATION PROGRAM FUEL MATERIALS ANALYSIS

Fuel Material	U ₃ O ₈	UAl ₃	UO ₂
Source	Y-12	B&W	NUMEC
Grade	Dead-Burned	----	Nb Coated
U (wt%)	84.67	69.11	51.82
U-235 (percent)	93.16	92.63	93.16
Al (ppm)	6	Balance	Not Sought
B (ppm)	< 1	Not Found	Not Sought
Ba (ppm)	3	Not Sought	Not Sought
Be (ppm)	< 0.1	Not Sought	Not Sought
C (ppm)	Not Reported	330	Not Sought
Ca (ppm)	20	Not Sought	Not Sought
Cd (ppm)	< 0.1	Not Sought	Not Sought
Co (ppm)	< 1	Not Sought	Not Sought
Cr (ppm)	6	240	Not Sought
Cu (ppm)	6	94	Not Sought
Fe (ppm)	40	740	Not Sought
Li (ppm)	0.5	Not Sought	Not Sought
Mg (ppm)	9	Not Sought	Not Sought
Mn (ppm)	< 1	31	Not Sought
Na (ppm)	3	Not Sought	Not Sought
Nb (wt%)	Not Sought	Not Sought	36.6
Ni (ppm)	6	250	Not Sought
O (ppm)	Balance	Not Sought	Balance
P (ppm)	< 100	Not Sought	Not Sought
Si (ppm)	10	180	Not Sought
Sn (ppm)	40	48	Not Sought
Ti (ppm)	Not Reported	2.5	Not Sought
V (ppm)	< 1	1	Not Sought
Zr (wt%)	Not Sought	Not Sought	2.0
X-ray Diffraction	----	"UAl ₃ is the major crystalline component. UAl ₂ and UAl ₄ are probably present."	----
Mesh Size	-170+325	-170+325	-200+230
Measured Density	8.33	6.27	----

- (1) The U_3O_8 shall contain no detectable UO_2 and shall have a density higher than 8.2 g/cm^3 . The particle size shall range between -170 to +375 mesh and shall not contain more than 10 percent of -325 particles. The total uranium concentration shall be a minimum of 84 weight percent. The surface area of the material shall not exceed $0.05 \text{ m}^2/\text{g}$.
- (2) The uranium-aluminum intermetallic compound powder was purchased from the Babcock and Wilcox Company and was to conform to the following specifications:
 - (a) The uranium-aluminum alloy powder used in the cores shall be made from highly enriched (93 percent U-235 minimum) uranium metal alloyed with aluminum alloy 1060, ASTM Specification B-209-62.
 - (b) The uranium content of the uranium-aluminum material shall be 70 ± 2.5 weight percent.
 - (c) It shall be heat treated so that the major crystalline constituent, as determined by X-ray diffraction, is UAl_3 .
 - (d) No uranium oxides, nitrides, carbides, or hydrides shall be present.
 - (e) The uranium-aluminum alloy shall be reduced to a powder having a particle size ranging from -170 to +325 mesh as determined by screening on U. S. standard sieves.

From the data in Table II, it can be seen that the material did not meet this specification with respect to the U-235 content which was slightly low.

The spheroidal, niobium-coated $\text{UO}_2\text{-ZrO}_2$ powder was supplied by the Nuclear Materials and Equipment Corporation at Apollo, Pennsylvania. Chemical and physical properties measured for the two batches of coated oxide are shown in Table II.

2.12 Burnable Poison. The B_4C powder conformed to the "high-purity" grade of the Norton Chemical Company and analyzed 75.9 percent natural boron. The major impurities were 0.1 percent Fe, 0.3 percent B_2O_3 , and traces (0.5 percent total) of Na, Ca, Al, Si, N, and H_2O . The particles were less than 44μ in diameter, as determined by screening on U. S. standard sieves.

2.13 X8001 Aluminum Powder. The X8001-Al powder was supplied by the Reynolds Metals Company and conformed to Grade 1-701 for -100 mesh atomized material. The following impurity-content limits applied: B, 0.001 percent max; Cd, 0.002 percent max; and Li, 0.008 percent max. This powder had been vacuum annealed by ORNL.

2.14 Picture Frame and Cladding Materials. The wrought material for the frames and cladding of the fuel plate was to conform to ASTM Specification, B-209-62, clad GS11A, with the following modifications:

- (1) Picture Frames -- Aluminum Plate Alloy 6061-F clad on both sides with Alloy 1100. The thickness of Alloy 1100 cladding

material on each side was to be 5 percent of the total thickness of the composite plate.

- (2) Cladding -- Aluminum Plate Alloy 6061-F clad on one side with Alloy 1100. Thickness of Alloy 1100 shall be 5 percent of the total thickness of the composite plate. All 1100 and 6061-F materials were to be low-boron grade. The following composition limits were to apply:

Cadmium	0.002 percent maximum
Lithium	0.008 percent maximum
Cobalt	0.001 percent maximum
Boron	0.001 percent maximum

In Table III, the chemical analyses of the materials are shown; and in Table IV, the screen analysis of X8001 aluminum powder is shown.

TABLE III

ATR FUEL IRRADIATION PROGRAM
ALUMINUM BASE MATERIALS ANALYSIS

Element as Determined by Spectral Analysis (percent)	Aluminum Alloy		
	1100	6061	X8001
B	< 0.001	< 0.001	< 0.001
Cd	< 0.005	< 0.005	< 0.005
Cr	0.0022	0.16	0.0018
Co	< 0.001	< 0.001	< 0.001
Cu	0.35	0.20	0.0035
Fe	0.35	0.43	0.28
Li	---	---	< 0.01
Mg	0.82	0.002	0.002
Mn	0.011	0.006	0.006
Ni	0.003	0.002	0.9
Pb	< 0.025	< 0.025	< 0.025
Si	0.60	0.11	0.06
Sn	0.096	0.009	0.009
Ti	0.024	0.01	0.0031
Zn	0.009	0.008	0.007

TABLE IV

SCREEN ANALYSIS OF
X8001 ALUMINUM POWDER

U.S. Standard Mesh Size	Percent
+ 80	0.3
+100	2.1
+140	9.5
+200	10.5
+230	5.9
+325	16.7
-325	55.0

2.2 Sample Dimensions

In Figures 5 and 6 are shown the details of the two types of sample fuel plates used in the irradiation experiment. The single-core test plates were considered to be the standard test plates in the irradiation experiment while the double-core plates were made for use as instrumented plates to provide information as to the temperatures achieved during the irradiation testing.

2.3 Fabrication Procedures

2.31 PPCo Samples. The fabrication of the single-core test plates was performed in accordance with the procedures given on the following page:

Fig. 5 PAED-59 experiment, ATR fuel development program, single-core test plate details.

Fig. 6 PAED-59 experiment, ATR fuel development program, double-core test plate details.

- (1) Fuel Core Fabrication -- The following steps were used in the fabrication of the cores:
 - (a) Receive X8001 aluminum powder, U_3O_8 powder, UAl_3 powder, and B_4C powder according to specification requirements. Store in vacuum desiccator until ready for use.
 - (b) Weigh out precise quantities of aluminum powder, fuel powder, and B_4C . A sufficient quantity of powders should be taken in each batch to make nine cores. Record weight of powders used for each batch.
 - (c) Blend powders for four hours.
 - (d) Divide blended powders into nine equal parts by weighing. Each of these parts is to contain the powder for one fuel

plate core. The nine fuel plate cores from each batch of powders are to be processed together as one assembly.

(e) Wipe compacting die and punches clean and apply a light coating of stearic acid-methanol solution. Pour core powders into compacting die and level powder.

(f) Compact powder using a pressure of 33 tons per square inch (tsi).

(g) Weight cores and record weights.

(2) Sample Fuel Plate Fabrication -- the steps to be used for fabrication of the sample fuel plates (Figure 7) were:

Fig. 7 Fabrication of ATR sample fuel plates.

(a) Receive 6061-F alclad (one side) aluminum alloy. Roll to required thickness for cover plates and cut into pieces measuring 6-1/2 by 7 inches.

(b) Receive 6061-F alclad (both sides) aluminum alloy. Roll into thickness required for picture frames and shear into pieces measuring 6-1/2 by 7 inches. Punch nine core cavities as shown in Figure 7.

(c) Clean picture frames by immersion in Oakite 160 solution, rinsing in deionized water, pickling in 50 percent HNO_3

solution, rinsing in water, rinsing in methanol, and drying with hot air.

- (d) Assemble the core compacts into the picture frames and install cover plates which are to be held in place by means of two rivets. (See Figure 8.)
- (e) Preheat the assemblies for $3/4$ hour at 500°C . Hot roll on a Fenn mill with 8-1/2-inch-diameter rolls using 20 percent reduction per pass and a 5-minute reheating time between each pass until a thickness of 0.059 inch is reached.
- (f) Blister anneal plates for 1 hour at 500°C . Halfway through the anneal, the plates are to be rotated end-for-end in the furnace. Inspect plates for blisters.
- (g) Radiograph plates to determine core length. Mark plates to indicate ends of cores.
- (h) Cold roll plates until cores are 5-1/2 inches long.
- (i) Section the plates by shearing through the middle of the two webs separating the three rows of plates. Mark the three pieces thus produced so as to maintain the identity of the cores. Anneal for two hours at 425°C . Halfway through the anneal, the plates are to be rotated end-for-end in the furnace.
- (j) Cover both sides of plates with masking tape and roll level.
- (k) Radiograph plates to locate cores. Shear plates to final size. Save the scrap for tensile specimens and hardness tests.
- (l) Remove masking tape and stamp identifying numbers on plates.
- (m) Determine Rockwell superficial hardness on the 15-T scale on the scrap from the plates. At least three readings which check to within ± 2.5 are made on the scrap from each set of the plates. Two tensile specimens are to be cut and pulled from the scrap from each set of three plates.
- (n) Clean fuel plates using procedure given in Step (c) above.

Fig. 8 Sample fuel plate rolling assembly ready to be put together and rolled.

The double-core test plates made by Phillips Petroleum Company were fabricated in essentially the same manner as that used for the single-core plates down to Step (e) of the sample fuel plate fabrication procedure. The exceptions being that only half as much core material was used, with the result

that the picture frame thickness was cut in half and that 1/16 inch was filed off the cores to allow for widening during the eutectic bonding operation. The assemblies were rolled until the core lengths (as determined by radiography) were 3-5/8 inches. At this point, the assemblies were approximately 0.06 inch thick. The assemblies were separated into individual fuel plates (measuring 1-1/2 by 5 inches) by shearing; and each of the plates was ultrasonically inspected to ensure bonding.

Two of the fuel plates were then bonded to an aluminum filler piece 0.120 to 0.125 inch thick using a eutectic bonding procedure similar to the technique developed by Walker and Noland of ANL^[4]. In this process, elemental silicon powder with the following particle size range was used:

<u>Mesh Range</u>	<u>Weight Percent</u>
-200 +325	84
-100 +200	16

The silicon was suspended in a slurry of 0.25 g/cc of silicon in solution of 67 volume percent of glycerin and 33 volume percent of ethyl alcohol. The slurry was applied to the inside faces (alclad) of the plates to be joined, as shown in Figure 9(a), and dried with a gas torch [Figure 9(b)]. Aquadag was applied to the outside surfaces of the fuel plates to prevent the plates from sticking to the heated platens. Figure 9(c) shows the three plates ready for pressing.

The coated faces were brought into intimate contact with each other, were placed between the heated platens of a press, and were maintained at temperatures above the melting point of the aluminum-silicon eutectic (571°C) and below the solidus temperature of 6061 aluminum alloy (582°C). Pressures of 950 to 1000 psi were maintained on the plates. Figure 10 shows the equipment used for this operation.

Heating was continued until beads of the aluminum-silicon alloy were visible all around the periphery of the assembly. At this time, the work was cooled, under pressure, to a temperature of 500°C. Then the press was opened, and the bonded plate was removed.

The plates were hot and cold rolled to produce a core length of 5-1/2 inches. Since the plates were too thick to be sheared to the final dimensions on the existing equipment in the laboratory, the plates were milled to size (after being radiographed), heat treated, and cleaned. Figure 11 shows a radiograph of a plate made in this manner. As can be seen, excellent control over the alignment of the cores could be achieved by this process. However, considerable difficulty was encountered in the spreading of the cores in the hot pressing operation, but decreasing the core compact width by 1/16 inch eliminated this problem.

2.32 ORNL Sample Fabrication Procedures^[a]. The essential operations required to fabricate the miniature fuel plates prepared by ORNL were as follows on page 22:

[a] Written by M. M. Martin and C. F. Leitten, Jr., Metals and Ceramics Division Oak Ridge National Laboratory, Oak Ridge, Tennessee.

64-4817

64-4819

(A) APPLYING SILICON SLURRY.

(B) DRYING OF SLURRY.

(C) DOUBLE CORE FUEL PLATE ASSEMBLY BEFORE PRESSING.

Fig. 9 Steps in preparing double-core fuel plates for pressing.

Fig. 10 Hot pressing and temperature control equipment used to make double-core plates.

Fig. 11 Radiographs of double-core plate.

- (1) Weighing and blending of the component powder for each fuel core
- (2) Pressing and, in some cases, machining of the fuel compacts to the designed dimensions
- (3) Degassing of the compacts
- (4) Assembling the cores within a welded billet
- (5) Cladding by roll-bonding
- (6) Annealing to soften and also test for blistering
- (7) Cold rolling to specified thickness
- (8) Heat treating to the "O" temper
- (9) Marking and shearing of the plate
- (10) Machining to finished length and width dimensions
- (11) Inspecting and final cleaning
- (12) Packaging for shipment.

The specific details for these general procedures are described below and represent many of the methods that were developed and adopted for manufacturing HFIR and ATR fuel plates[5,6].

(1) Component Design. The billets for the sample and instrument plates contained two cores which were separated by a distance of 2 inches in the direction parallel to rolling to permit the simultaneous fabrication of two plates. The cores also were separated in the instrument billet but were separated in the direction normal to rolling. Thus, one billet was required for each instrument plate.

Design dimensions for the sample and instrument billets are given in Table V; the table also includes a list of the components and a description of the materials. The small differences in length and width of the fuel compacts, which are reflected in the cavity dimensions, are the result of different estimates of spread and densification for the various core compositions.

(2) Compact Preparation. Although close control of uranium content per plate dictates the preparation of powders for each fuel section, limitations of time and available equipment necessitated the partial use of master fuel compacts. Individual core sections from the master compacts were machined.

Conventional powder metallurgical techniques were employed to blend and press individual fuel compacts of composition D and master fuel compacts of compositions B and C (see Table VI). Precise quantities of the component powder (as shown in Table VII) were weighed separately, combined in a blending jar, blended for 2 hr without a binder addition, and immediately compacted at a pressure of 22.5 tons per square inch to highly dense rectangular shapes. The faces of the powder metallurgy die were lubricated with a solution of chemically

TABLE V
SPECIFICATIONS FOR INSTRUMENT AND SAMPLE BILLETS

Type of Billet	Composition	Components of Billets	Number Required	Materials	Dimensions (in.)		
					Length	Width	Thickness
Sample	B	Cover Plate	2	Alclad 6061-F Al	7.000 ± 0.015	5.000 ± 0.015	0.120 ± 0.001
Sample	B	Picture Frame	1	Alclad 6061-F Al	7.000 ± 0.015	5.000 ± 0.015	0.157 ± 0.001
Sample	B	Cavity ^[a]	2	-----	0.742 ± 0.002	0.957 ± 0.002	-----
Sample	B	Fuel Compact ^[a]	2	42 percent U ₃ O ₈ , fully enriched, plus X8001 Al	0.743 ± 0.001	0.960 ± 0.001	0.160 ± 0.001
Instrument	C	Cover Plate	2	Alclad 6061-F Al	7.000 ± 0.015	5.000 ± 0.015	0.115 ± 0.001
Instrument	C	Split-Core Frame	1	Type 6061-T6 Al	7.000 ± 0.015	5.000 ± 0.015	0.926 ± 0.002
Instrument	C	Cavity ^[a]	2	-----	0.719 ± 0.001	0.957 ± 0.001	0.0780 ± 0.0005 ^[b]
Instrument	C	Fuel Compact ^[a]	2	43 percent U ₃ O ₈ , fully enriched, plus X8001 Al	0.720 ± 0.001	0.960 ± 0.001	0.081 ± 0.001
Sample	D	Cover Plate	2	Alclad 6061-F Al	7.000 ± 0.015	5.000 ± 0.015	0.123 ± 0.001
Sample	D	Picture Frame	1	Alclad 6061-F Al	7.000 ± 0.015	5.000 ± 0.015	0.157 ± 0.001
Sample	D	Cavity	2	-----	0.733 ± 0.002	0.961 ± 0.002	-----
Sample	D	Fuel Compact	2	56 percent Nb-coated UO ₂ -ZrO ₂ , fully enriched U, +0.16 percent B ₄ C plus X8001 Al	0.733 ± 0.001	0.964 ± 0.001	0.160 ± 0.001

[a] 1/32-inch radius on corners of frame cavity and fuel compact.

[b] Depth of depression.

TABLE VI
CORE COMPOSITION FOR SAMPLE AND INSTRUMENT PLATES

ORNL Fabrication Run	Type of Plate	Core Material ^[a]
B	Sample	42 percent U ₃ O ₈
C	Instrument	43 percent U ₃ O ₈
D	Sample	56 percent Nb-coated UO ₂ -ZrO ₂ plus 0.16 percent B ₄ C ²

[a] Balance -- Type X8001 Al.

TABLE VII

WEIGHT OF COMPONENT POWDERS FOR FUEL COMPACTS

Composition	Die Size (in.)	Weight [a] (g)			
		U_3O_8	Nb-Coated UO_2-ZrO_2	B_4C	X8001 Al
B	1.000 x 1.800	7.108	---	---	9.840
C	1.000 x 1.800	3.665	---	---	4.866
D	0.733 x 0.964	---	4.570	0.0128	3.620

[a] To ± 0.001 g.

pure stearic acid in carbon tetrachloride. Before the master compacts were machined and the fuel sections were assembled in the billets, the volatile constituents of the lubricant were removed by heating the compacts 2 hr at 550°C at a pressure less than 0.1 millimeters of mercury (torr). Two rectangular core sections were then machined from each master compact. The corners of the core sections for the instrument plates and the sample plates that contain Y-12 U_3O_8 were filed to a radius of 1/32 inch.

(3) Billet Assembly. As illustrated in Figure 7, each fuel plate billet required two cover plates and one frame in addition to the two fuel compacts. Cover plates were cold rolled from 0.16-inch-thick sheet and sheared. The frames for the sample plates were fabricated from 0.425-inch-thick plate by hot rolling to an intermediate thickness of 0.2 inch, by cold rolling to a finish thickness of 0.157 ± 0.001 inch, and by machining. The corners of the two windows in the frames were broached square for fuel sections of compositions A and D, and those for fuel sections of composition B were machined to include 1/32-inch radii. The instrument-plate frames contained top and bottom depressions, with 1/32-inch radii at the corners, for accepting the fuel sections. These frames were machined from 1-inch-thick wrought plate.

To determine the integrity of the cladding materials and of the alclad bond, the cover plates and frames were heated to 500°C in air for at least 85 minutes. Pieces that blistered were rejected.

Immediately prior to billet assembly, both surfaces of the frames and the alclad surface of each cover plate that contacts the frame were thoroughly cleaned by scratch brushing with a power-driven stainless steel brush. To provide an interference fit, the width and length of the compacts were designed to be essentially identical to the dimensions of the cavity in the frame. To assemble these components, the frame was heated to 500°C, the cold compacts were slipped into the expanded frame cavities, and then the frame was allowed to cool to

room temperature. To provide excess material for densification during roll cladding, the compacts were 0.003 inch thicker than the frame.

The cover plates were attached to the edges of the frame by tungsten inert-gas welding. High-quality welds were not required since the welds were needed only to prevent shifting of the billet components during the initial stage of the hot-rolling operation.

(4) Composite Plate Fabrication.

(a) Hot Rolling. The assembled and welded billets were hot rolled in lots of four on a two-high Mesta Mill equipped with 20-inch-diameter by 30-inch-wide rolls. The roll faces were well lubricated with a mixture of one part SAE 90-weight oil to two parts kerosene at all times during hot rolling. Immediately after being welded and before the first mill pass, the billets were heated to $500 \pm 10^\circ\text{C}$ on a clean furnace hearth of stainless steel. The sample billets were placed in the furnace with either the top or bottom cladding in contact with the hearth. The billets for the instrument plates, however, were supported on their edges during preheating and reheating to ensure a uniform temperature through the thickness of the billets, thus allowing equal amounts of work to be imparted to the two cores during rolling.

The hot-reduction schedules, based on mill settings, and the hot-finished thicknesses of the sample and instrument billets are shown in Table VIII. Near the end of the schedules, the reduction per pass were reduced successively to minimize thickness variations within a plate. All billets were rotated 180 deg about their longitudinal and transverse axes and reheated between passes. On the final mill setting, the plates were fed twice through the mill without reheat, and their thicknesses were immediately measured with a micrometer. Additional double passes were made, as required, to roll the sample plates to 0.0625 ± 0.0005 inch and the instrument plates to 0.187 ± 0.001 inch.

TABLE VIII

HOT REDUCTION SCHEDULES FOR
SAMPLE AND INSTRUMENT BILLETS^[a]

Mill Setting	Reduction (percent)	
	Sample Billet	Instrument Billet
1	15.0	14.5
2	15.0	14.5
3	22.5	14.5
4	22.5	14.5
5	22.5	14.5
6	22.5	14.5
7	22.5	14.5
8	12.0	14.5
9	8.5	14.5
10	5.5	14.5
11	----	14.5
12	----	7.0
13	----	3.5

[a] Conditions: Furnace temperature -- $500 \pm 10^\circ\text{C}$; billet preheat -- sample, 45 min and instrument, 100 min; reheat per pass -- sample, 5 min and instrument, 15 min. through mill setting No. 4, 5 min thereafter.

After cooling, the hot-rolled plates were numbered consecutively, starting with 800 for the first plate processed. The consecutive plate numbers were prefixed with 0-3 and 0-4 to identify the ORNL fuel plate containing U_3O_8 and coated $\text{UO}_2\text{-ZrO}_2$, respectively. The numbers, which were 1/2 inch high, were stamped on each plate in the inactive section at the end that had been fed first through the mill. Prior to annealing, the plates were degreased in trichlorethylene vapor and sheared to remove approximately 2 inches of excessive aluminum from the ends. The rolled sample billets were also sheared in half to separate the two fuel plates.

(b) Softening Anneal. To remove stresses induced in the last few passes of the hot-rolling process and to test for blisters, the fuel plates were annealed for 1 hr at 500°C and then air-cooled to room temperature. Plates that contained one or more blisters were rejectable. No plate exhibited blisters after either this anneal or the subsequent final heat treatment.

(c) Cold Rolling. After visual inspection for blisters, the plates were cold rolled 20 percent on a two-high United Mill equipped with 12-inch-diameter by 14-inch-wide rolls that were well lubricated with a mixture of two parts of SAE 90-weight oil to one part kerosene. The entering ends of the plates were dipped into the oil mixture, as required, before insertion into the mill. At the completion of cold rolling, the plates were degreased in trichlorethylene vapor. With the core approximately centered, the ends were then sheared to yield a plate 12.0 inches long by 5.2 inches wide.

The cold-reduction schedules, based on mill settings, were identical for the sample and instrument plates. The first setting was for approximately 8.5 percent reduction, the next three were for 3.5 percent each, and the final setting was for 2.0 percent. Four passes were made at each mill setting; between passes, the plates were rotated 180° about their longitudinal and transverse axes to minimize thickness variations, which in any one plate were within ± 0.0002 inch. The cold-finished thicknesses for the sample and instrument plates were 0.050 ± 0.001 inch and 0.150 ± 0.001 inch, respectively.

(d) Final Heat Treatment. To facilitate subsequent marking, machining, and assembly of the irradiation test elements, the composite plates must be flat within ± 0.003 inch and free of stresses induced in rolling. The plates were flattened and annealed at 500°C under a static load of 0.3 lb/in.^2 in the final heat treatment. Two 1-inch-thick platens of Type 6061 aluminum were used in this operation. In contrast to flat annealing of stainless steel, the aluminum clad fuel plates were not clamped in place since flattening was achieved through the weight of the top platen. The plates were assembled in two stacks of equal height between the platens and inserted into a top-loading furnace at room temperature and heated to 500°C ; the plates were heated at a rate of approximately 200°C/hr . After annealing for 2 hr at 500°C , the plates were furnace-cooled to room temperature at a rate of 25°C/hr and again inspected for blisters. Plates that contained one or more blisters were rejectable.

(e) Examination and Marking. After annealing and visual inspection, the plates were fluoroscoped with a 175-kvp machine to delineate the

location of the fuel-bearing section. Lines for cutting the plates to 1-7/8 inches wide by 6-3/8 inches long (with the core approximately centered) were scribed on the numbered surface of each plate. The fuel plate numbers were then inscribed with a Burgess Vibro-tool at a location just inside the scribe line at the same end of the plate.

The sample plates were sheared on the scribe lines. The ends and sides of the instrument plates were sawed off to avoid the possibility of breaks that may occur between the cladding and the core during the shearing of thick fuel plates near the core edge. After being sized, the plates were machined to establish two reference edges, at right angles to each other, for locating the position of the core to a precision of ± 0.005 inch. Approximately 1/8 inch of excessive aluminum was removed from the width and length of the plates. The position of the core was then determined from radiographs. A vernier caliper was used to measure accurately the location with respect to the machined edges. Prior to machining, the fuel plate's number was again inscribed with the same identification and by the same tool in numerals about 3/32 inch high at the same plate end.

(f) Machining. The samples and instrumented plates were machined individually on a vertical milling machine equipped with a side-cutting tool. First, each composite plate was sandwiched between 1/8-inch-thick aluminum backup plates of the same length and width. After the reference edges were aligned with the milling tool and the assembly firmly fixed on the milling machine table with hold-down clamps, the longitudinal sides of the plates were machined to within approximately 0.125 inch of the core to yield a finish plate width of 1.250 ± 0.003 inches. The clamps were then repositioned, as required, to machine the plate to final length of 5.750 ± 0.003 inches. Approximately equal amounts of aluminum were removed from each edge of the fuel plates. With the fuel plate number up, a 1/16-inch by 45 deg chamfer was then milled on the numbered end in the right-hand corner of the plate. After completion of the machining operation, the plates were degreased in trichlorethylene, and the machined edges were lightly deburred by hand filing.

(g) Inspections. All plates were inspected to ensure that bonding, specified total uranium content, and the specified dimensions in machining and rolling had been achieved. The Nondestructive Testing Group evaluated the plates for continuity of ultrasound transmission, fuel homogeneity, and fuel loading. The length and width of the cores were measured from radiographs and recorded along with the positions of the cores with respect to the overall dimensions of the machined plates. The weights, thicknesses, and pycnometric densities of the fuel compacts and of the finished plates after pickling were measured. The average core thickness was then calculated from actual spread, densification, and elongation of the core.

(h) Preparation for Shipment. Before and after the density determination, all plates were pickled at room temperature for 2 min in a solution of 1 part 60 percent HF, 10 parts concentrated HNO₃, and 100 parts H₂O by volume and were rinsed in warm water (100°F) for 3 to 5 min. Approximately 0.0001 to 0.0003 inch of aluminum was removed from the plate thickness in this final operation. The plates were then wrapped individually in tissue paper to prevent surface damage and packed in plywood boxes for shipment.

2.4 Sample Inspection Methods

After fabrication, the sample fuel plates were subjected to a series of tests designed to determine their suitability for irradiation in the ETR and to establish their metallurgical properties prior to irradiation so that the irradiation effects could be evaluated. The following properties of the fuel plates were measured: dimensions, weight, uniformity of fuel distribution in the core, location of the core, tendency for blister formation in the cladding, hardness of the core and cladding, thickness of the cladding, the quality of the bonds between the core, and the cladding and the density of the plates. In order to measure these properties, the following tests were performed:

- (1) Blister Test -- this was done by annealing the plates at 500°C for one hour before the final cold-rolling passes and then carefully inspecting the surfaces for blisters.
- (2) Visual Inspection -- the plates were inspected to ensure that they were completely free of dirt, scale, grease, or other foreign materials. Pits and other surface defects which were less than 0.005 inch deep were permitted.
- (3) Dimensional Check -- the dimensions of the fuel plates were checked with a micrometer and a venier caliper.

The thickness was measured at each corner over the core and in the center of the plate. The width was checked at each end and in the middle of the plate. The length was measured along each edge.

The irradiation sample specifications were as follows:

	Core Dimensions	
	Single-Core Plates	Double-Core Plates
Length	5.750 ± 0.005 inches	5.750 ± 0.005 inches
Width	1.250 ± 0.005 inches	1.250 ± 0.005 inches
Thickness	0.050 ± 0.005 inch	0.150 ± 0.005 inch

- (4) Gross Weight -- the samples were weighed on an analytical balance with a precision of 0.1 mg.
- (5) Density Determination -- this test was done by water immersion before and after irradiation using ASTM Procedure B328-58T.
- (6) Radiographs -- nine plates from each fabrication batch were radiographed at one time. The radiographs of the core areas were scanned with a densitometer to ensure that the fuel from each blending batch was uniformly distributed among the nine fuel plate cores in each fabrication batch of plates. The radiographs were inspected to ensure that there was a uniform distribution of fuel in each plate. The length and width of the

cores were measured and the area determined. The radiographs were inspected to ensure that the core dimensions were in accordance with the following:

	Core Dimensions	
	Single-Core Plates	Double-Core Plates
Length	5-7/16 $\pm$ 3/32 inches	5-3/8 $\pm$ 1/8 inches
Width	1 $\pm$ 1/32 inch	1 $\pm$ 1/16 inch
Thickness	0.020 $\pm$ 0.003 inch	0.010 $\pm$ 0.002 inch (each half core)

- (7) Core Thickness and Density -- from the gross weights, core weights, plate densities, and cladding material densities, the core densities were calculated. By use of these data as well as the core areas as determined by radiography, the core thickness of the plates was determined.
- (8) Whole Plate Alpha Counting -- the plates were placed inside the chamber of an alpha counter and inspected to ensure no contamination existed on the surfaces.
- (9) Ultrasonic Inspection -- the samples were inspected by immersion testing, using the through-transmission technique. Five mc Li_2SO_4 crystals were used with suitable standards to ensure that nonbonds 1/8 inch in diameter or larger could be detected.
- (10) Liquid Nitrogen Testing -- the samples were submerged in liquid nitrogen until bubbling stopped. They were then quickly transferred to a bath of methyl alcohol. Any crack would have been revealed by a stream of bubbles.
- (11) Destructive Examination -- representative plates were destructively examined in order to determine the preirradiation microstructure and microhardness of the constituents. Several plates were dissolved in order that the total uranium, U-235, and boron content could be checked against the calculated amounts. On the Phillips plates, tensile tests were performed on the scrap aluminum trimmed from the rolled and heat-treated assemblies in order to ensure that the cladding and picture frame alloy was in the fully annealed ("O") condition.

2.5 Inspection Results

The pertinent data obtained on the weights, densities, and dimensions of all the irradiation specimens are recorded in Table IX. The mechanical properties of the picture frame and cladding materials of the plates fabricated by Phillips Petroleum Company are given in Table X and indicate that the 6061 aluminum in the samples was in the "O" condition.

In Table XI a comparison is made of the uranium, U-235, and boron contents of sample plates as calculated from the core weights and compositions and as

TABLE IX

PARAMETERS OF ATR FUEL IRRADIATION PROGRAM SPECIMENS

Composition Number	Plate Number	Fabricator	Number of Cores	Sample Fuel Plate					Core Weight (g)	U-235 Weight (g)	B ₁ C Weight (g)	Average Core				
				Length (in.)	Width (in.)	Thickness (mils)	Gross Weight (g)	Density (g/cc)				Calculated Density (g/cc)	Length (in.)	Width (in.)	Area (in. ²)	Calculated Thickness ^[a] (mils)
1	584	PPCo	1	5.751	1.246	51.4	17.779	2.996	6.742	2.212	0.0128	3.636	5.435	0.995	5.408	20.9
1	585	PPCo	1	5.751	1.250	51.3	17.854	2.996	6.749	2.211	0.0128	3.640	5.461	0.993	5.423	20.9
1	587	PPCo	1	5.750	1.249	51.1	17.795	2.996	6.739	2.211	0.0128	3.637	5.445	0.994	5.412	20.9
1	588	PPCo	1	5.752	1.249	51.3	17.846	2.997	6.741	2.212	0.0128	3.644	5.449	0.991	5.433	20.8
1	589	PPCo	1	5.752	1.250	51.0	17.784	2.993	6.747	2.214	0.0128	3.624	5.500	1.002	5.511	20.6
1	651	PPCo	2	5.748	1.249	154.0	50.113	2.79	6.719	2.204	0.0128	3.50	5.400	1.043	5.632	10.4
1	653	PPCo	2	5.748	1.249	151.0	49.496	2.79	6.734	2.209	0.0128	3.49	5.365	1.044	5.601	10.5
1	654	PPCo	2	5.749	1.250	152.0	49.767	2.79	6.698	2.198	0.0127	3.50	5.275	1.036	5.465	10.7
1	673	PPCo	2	5.743	1.247	149.0	48.444	2.796	6.724	2.206	0.0128	3.53	5.291	1.026	5.429	10.7
1	674	PPCo	2	5.742	1.247	149.0	46.906	2.796	6.683	2.193	0.0127	3.51	5.347	1.032	5.518	10.6
1	676	PPCo	2	5.743	1.247	147.0	48.058	2.796	6.713	2.203	0.0127	3.53	5.350	1.041	5.569	10.4
2	593	PPCo	1	5.748	1.249	51.7	17.791	2.997	6.729	2.208	0.0128	3.643	5.472	0.999	5.467	20.6
2	598	PPCo	1	5.749	1.251	51.6	17.830	2.974	6.715	2.203	0.0128	3.560	5.430	1.000	5.430	21.2
2	599	PPCo	1	5.751	1.252	51.5	17.864	2.981	6.732	2.209	0.0128	3.585	5.450	0.994	5.417	21.2
2	601	PPCo	1	5.749	1.250	51.5	17.791	2.939	6.715	2.203	0.0128	3.430	5.520	0.990	5.465	21.9
2	614	PPCo	1	5.747	1.253	52.2	18.306	2.99	6.728	2.207	0.0128	3.612	5.340	1.007	5.377	20.9
2	655	PPCo	2	5.748	1.250	147.0	48.283	2.80	6.741	2.212	0.0128	3.57	5.337	1.019	5.438	10.6
2	658	PPCo	2	5.747	1.249	152.0	49.835	2.79	6.720	2.205	0.0128	3.49	5.480	1.050	5.754	10.2
3	803	ORNL	1	5.751	1.250	50.7	17.734	2.991	6.657	2.202	0	3.630	5.484	1.003	5.500	20.3
3	805	ORNL	1	5.751	1.250	50.7	17.740	2.990	6.617	2.189	0	3.634	5.483	1.000	5.483	20.3
3	806	ORNL	1	5.751	1.250	50.7	17.636	2.986	6.594	2.181	0	3.615	5.484	1.000	5.484	20.3
3	812	ORNL	1	5.750	1.250	50.5	17.648	2.986	6.612	2.187	0	3.613	5.470	1.002	5.481	20.4
3	815	ORNL	1	5.749	1.250	50.2	17.581	2.991	6.581	2.177	0	3.634	5.472	0.996	5.450	20.3
3	825	ORNL	2	5.748	1.250	149.0	N.D.	N.D.	6.563	2.200	0	N.D.	5.353	1.000	5.353	N.D.
3	827	ORNL	2	5.750	1.249	149.0	N.D.	N.D.	6.553	2.213	0	N.D.	5.484	1.001	5.489	N.D.
4	843	ORNL	1	5.752	1.251	50.5	19.245	3.253	8.178	2.199	0.0128	4.481	5.398	1.006	5.430	20.5
4	844	ORNL	1	5.752	1.251	50.5	19.212	3.253	8.152	2.192	0.0127	4.487	5.373	1.009	5.421	20.5
4	848	ORNL	1	5.751	1.250	50.4	19.154	3.256	8.169	2.197	0.0127	4.484	5.343	1.004	5.364	20.7
4	852	ORNL	1	5.751	1.250	50.3	19.087	3.259	8.166	2.196	0.0127	4.487	5.357	1.008	5.400	20.6
5	621	PPCo	1	5.745	1.250	53.0	18.553	2.99	6.730	2.424	0.0128	3.670	5.405	1.040	5.621	19.9
5	622	PPCo	1	5.745	1.250	53.0	18.524	2.98	6.746	2.429	0.0128	3.623	5.392	1.011	5.451	20.8
5	623	PPCo	1	5.747	1.251	52.7	18.390	2.99	6.735	2.425	0.0128	3.656	5.439	1.017	5.531	20.3
5	625	PPCo	1	5.744	1.252	52.6	18.431	2.99	6.745	2.429	0.0128	3.658	5.449	1.020	5.558	20.2
5	659	PPCo	2	5.747	1.249	153.0	49.651	2.80	6.737	2.426	0.0128	3.61	5.386	1.008	5.429	10.5
5	662	PPCo	2	5.748	1.249	152.0	49.817	2.80	6.736	2.425	0.0128	3.61	5.382	1.038	5.587	10.6

[a] Calculated from core area as determined by radiography and the immersion density of plate.

determined by analytical chemical procedures. The calculated U-235 contents tended to be slightly lower than the measured values. However, the nuclear calculations in the experiment were based on the calculated values.

Representative microstructures of the fuel plates which were destructively examined are shown in Figures 12 through 16. In Table XII, the average core thickness measurements as determined by direct measurements at 3/32-inch intervals on longitudinal and transverse sections of fuel plates are compared with the calculated core thickness of the same plates. The calculations provide a nondestructive method of determining the core thickness and density before and after irradiation. The calculated core thickness values are in poor agreement with the measured thickness.

TABLE X

MECHANICAL PROPERTIES OF PICTURE
FRAME AND CLADDING MATERIALS

Composition Number	Plate Number	Superficial Hardness 15-T	Ultimate Tensile Strength (psi)	Percent Elongation 2-inch Gages
1	584	27	15,400	21
1	585	27	15,400	21
1	587	25	15,400	20
1	588	25	15,400	20
1	589	25	15,400	20
2	593	25	15,400	21
2	598	26	15,500	22
2	599	25	15,400	21
2	601	25	15,400	21
2	614	47	18,000	16
5	621	48	19,500	17
5	622	48	19,500	17
5	623	48	19,500	16
5	625	48	19,500	16

NOTE: The tests were performed on scraps trimmed from the sample plates after heat treatment. The ASTM specification for aluminum alloy sheet and plate No. B209-59T requires that alclad 6061-0 aluminum alloy sheet, 0.021 to 0.128 inch thick, have a maximum ultimate tensile strength of 20,000 psi and a minimum elongation of 16 percent in a 2-inch test specimen.

TABLE XI

TOTAL URANIUM, U-235, AND BORON
CONTENTS OF SAMPLE FUEL PLATES

Composition Number	Plate Number	Total U		Total U-235		Total B	
		Calculated (grams)	Measured (grams)	Calculated (grams)	Measured (grams)	Calculated (grams)	Measured (grams)
1	586	2.388	2.460	2.212	2.285	0.097	0.0101
2	594	2.372	2.410	2.209	2.253	0.097	0.0101
3	820	2.351	2.33	2.190	2.17	0	0.001
4	836	2.395	---	2.232	---	0	0.001
5	606	2.612	2.635	2.420	2.447	0.097	0.0102

M-2578

AS POLISHED

500X

M-2586

AS POLISHED

50X

TRANSVERSE SECTION

Fig. 12 Typical microstructure of Composition 1 fuel plate before irradiation.

Fig. 13 Typical microstructure of Composition 2 fuel plate before irradiation.

Fig. 14 Typical microstructure of Composition 3 fuel plate before irradiation.

TRANSVERSE SECTION

Fig. 15 Typical microstructure of Composition 4 fuel plate before irradiation.

M-2887

AS POLISHED

500X

M-2888

AS POLISHED

50X

TRANSVERSE SECTION

Fig. 16 Typical microstructure of Composition 5 fuel plate before irradiation.

III. EXPERIMENTAL RESULTS

1. EVALUATION OF SAMPLE PERFORMANCE

The post-irradiation examination procedures are reported in this section, and the data obtained are compared to preirradiation sample inspection data for the purpose of evaluating fuel sample performance. Since the fuel materials were not irradiation tested to the point of decisive, visible, selective failure, as in a previous test^[7], it was necessary to probe the more subtle changes to assess the effects of swelling, incompatibility, corrosion, and damage by radiation and fission. The evidence needed for performance evaluation was found in comparative density data, microstructural changes, and mechanical property changes.

The approach that was used for the evaluation will be discussed briefly to serve as an orientation for Sections III-1.1 through III-1.7. A comparison of observed swelling with that amount expected from the various theoretical swelling mechanisms has shown gas-pressure-induced creep or yielding was not operative. Voids appearing in the post-irradiation microstructures had to be produced by other means. An accounting of swelling and void production was made by considering the void sources: fabrication, incompatibility, and the production of gaseous atoms. Since gas pressure in the voids was insufficient to cause creep or yielding of the cores, interconnecting of voids is necessary before blistering will occur. The necessary degree of interconnecting was calculated for a worst case. Two interconnecting mechanisms are demonstrated by the test (matrix cracking for the UO_2 and incompatibility for the U_3O_8), but neither mechanism produced in-pile blistering during the ETR Cycle 68 and 69, G-12 test series. The UAl_3 compositions did not exhibit an interconnecting mechanism.

Although corrosion metallography is presented in Section III-1.7, the problems created by an aluminum oxide film buildup are discussed in Section III-2.

1.1 Post-Irradiation Examination

The post-irradiation examination consisted of determining fuel plate physical and mechanical properties, metallography, qualitative analysis, and isotopic analysis. Figure 17 is a flow sheet showing how the samples were used in the different steps. The data were taken at the ETR canal, the TRA Hot Cell, and ICPP Analytical Chemistry Laboratory.

The physical properties taken include a record of plate appearance (ie, black and white and color photographs), coating thickness measurements, dimensional surveys, densities before and after film-stripping, and a core thickness survey.

Metallography was performed on each specimen to determine the progression of change with exposure, to study the effects of blister-anneal tests, to check the soundness of eutectic bond in the thermocouple plates, and to study the nature of the oxide films.

Fig. 17 Flow sheet of hot cell examination of ATR fuel irradiation samples.

A sample of the aluminum oxide coating was analyzed quantitatively, and U-235 isotopic burnup analyses were performed on 21 plates.

The pertinent portions of the voluminous data collected, which include over 400 photographs and over 1500 measurements, are reported in reduced form and are used to illustrate performance variables.

1.2 Core Volume Increases

Two approaches were used to determine the core volume changes resulting from irradiation: core thickness measurements and density measurements. The first method consisted of taking a large number of core thickness measurements and attempting to use these to determine core volume changes (see Table XII). The data, so derived, were very scattered and could not be interpreted. There were two basic problems which caused the scatter in the core thickness data. First, it was, of course, impossible to measure the core thickness metallographically of a fuel plate prior to irradiation. Consequently, the preirradiation thickness must either be calculated or measured on a companion sample which was rolled at the same time as was the irradiation specimen. Second, the cores of many of the sample fuel plates were very uneven so that wide spreads in maximum and minimum thickness values were obtained. Even though five

TABLE XII

COMPARISON OF FUEL PLATE CORE THICKNESS
AS DETERMINED BY DIRECT MEASUREMENT AND BY CALCULATION

Plate Number	Core Thickness (in.)		Percent Difference
	Calculated	Measured	
P-1-592	0.0215	0.0212	- 1.5
P-2-568	0.0202	0.0187	- 7.5
P-2-600	0.0213	0.0214	- 0.5
O-3-804	0.0200	0.0214	+ 7.0
O-3-874	0.0208	0.0209	+ 0.5
O-4-837	0.0193	0.0215	+11.4
P-5-573	0.0210	0.0208	- 1.0
P-5-608	0.0203	0.0211	+ 3.5
P-2-616	<u>0.0205</u>	<u>0.0198</u>	<u>- 3.5</u>
	Avg 0.0206	0.0207	+ 0.5

metallographic specimens were prepared from each irradiated sample, these still represent a rather small percentage of the total core volume.

By the second method, it is possible to determine core volume measurements to an accuracy better than 0.3 percent employing Archimedes' principle if the corrosion film is electrolytically stripped from the plate after irradiation. Then known quantities of solid aluminum (known weight and density) and of core material (known weight) are involved. The volume of the plate minus the volume of solid aluminum is the volume of the core. The observed percent change in core volume is given by the following equation:

$$\text{Percent } \frac{\Delta V}{V} (\text{obs}) = \frac{\left[V\text{-plate post} - \left(\frac{W\text{-plate post} - W\text{-core pre, post}}{\rho_{Al}} \right) \right] - \left[V\text{-plate pre} - \left(\frac{W\text{-plate pre} - W\text{-core pre, post}}{\rho_{Al}} \right) \right]}{V \text{ core pre}} \times 100$$

where

V is plate volume or core volume

W is plate or core weight

ρ_{Al} is aluminum density (2.705 g/cc)

subscript pre -- preirradiation

and

subscript post -- post-irradiation.

The core volume change values were also determined for the plates which were not stripped by making a correction for corrosion coatings. Although these values are not as accurate, they give statistical support to the value for the stripped plates. Both sets of data are shown in Table XIII.

Figure 18 presents the percent change in core volume with radiation exposure giving preference to the stripped plate determinations.

Fig. 18 Core volume behavior as a function of fission density.

TABLE XIII

THE PERCENT CHANGE IN VOLUME OF THE VARIOUS
FUEL PLATES WHICH WERE IRRADIATED TO VARIOUS EXPOSURES

<u>For Stripped Plates</u>				
<u>Composition Number</u>	<u>Plate Number</u>	<u>Irradiation Tier</u>	<u>10^{20} Fission/cc</u>	<u>Observed Percent $\frac{\Delta V}{V}$ (obs)</u>
1	584	3	7.3	0.6
1	587	4	16.3	5.1
2	598	4	16.2	7.3
2	599	5	6.3	0.2
3	812	6	3.8	0.5
4	844	4	17.0	11.8
<u>For Plates Which Were Not Stripped</u>				
<u>Composition Number [a]</u>	<u>Plate Number</u>	<u>Irradiation Tier</u>	<u>10^{20} Fission/cc</u>	<u>Observed Percent $\frac{\Delta V}{V}$ (obs)</u>
1	585	3	10.7	0.8
1	588	5	15.2	5.1
1	589	1	8.3	3.8
2	593	3 & 5	14.7	8.8
2	601	3	10.7	-1.6
2	614	1	8.4	0.5
3	803	6	6.7	3.5
3	805	1	7.5	-0.6
3	806	6	11.2	3.1
4	843	3	7.7	4.4
4	848	5	14.0	8.1
4	852	3	11.3	4.2
5	621	2	12.6	2.5
5	622	2	8.6	1.0
5	623	2	12.1	2.5
5	625	2	5.1	1.5

[a] The dispersed fuel material for composition Nos. 1 and 5 is UAl_3 ; for compositions 2 and 3, it is U_3O_8 ; and for composition No. 4, Nb-coated UO_2-ZrO_2 .

The basic swelling mechanisms considered are the following:

- (1) Atomic volume increase caused by uranium fission products
- (2) Vacancy absorption
- (3) Creep or yielding of the core.

At temperatures below $\approx 750^\circ\text{F}$, it has been shown that the principal mechanism which causes volume increases in metallic fuels is the change in atomic volume which occurs when uranium atoms fission[8, 9]. The value obtained for the percent volume has been referred to as " R_N^* ". For uranium, a volume increase of 3 percent for each 1 percent uranium burnup occurs if all the fission products are taken into account or 2.3 percent for each 1 percent uranium burnup if only those fission products that are solid are considered. The expected percent change in volume in a fuel plate core is given by

$$\text{Percent } \frac{\Delta V}{V} (\text{exp}) = \frac{W_{\text{U-235}} \times 0.03 \times \text{percent burnup} \times 100}{18.6 \times V_{\text{core}}} \quad (1)$$

The portion of this which could appear as void if all the fission gas is collected in voids (and swelling from gas pressure is discounted) is given by

$$\text{Percent } \frac{\Delta V}{V} (\text{fiss gas}) = \frac{0.7}{3.0} \times \text{percent } \frac{\Delta V}{V} (\text{exp}) \quad (2)$$

The importance of vacancy absorption would be realized only if voids increase beyond a critical size (≈ 1 micron). If the voids remain smaller than the critical size, the small volume change caused by vacancy diffusion is given by[10]

$$\frac{\Delta V}{V} = (KCTL/\gamma)^{3/2}$$

where

K = constant

C = concentration of fission product gases

T = absolute temperature

L = bubble spacing

and γ = surface tension.

Swelling by this mechanism is small because Van der Waal forces prevent ideal gas behavior for bubble diameters less than about 0.1 micron; between about 0.1 and 1 micron, the internal gas pressure is in equilibrium with surface tension forces[11]. The magnitude of swelling expected by this mechanism has not been assessed here; however, it is expected to be greater than extrapolated values of "R_N*" since the swelling is proportional to the 3/2 power of the gas concentration.

When the gas bubble size exceeds about 1 micron (either by continued diffusion of vacancies or by coalescence of gas bubbles or by diffusion of gas into the voids), the equilibrium between gas pressure and surface tension is nullified. Swelling of the gas bubbles under these conditions would be resisted by the yield strength or creep strength of the core material. If gas pressures are sufficiently high to cause creep or yielding of the material, the volume increases expected would be orders of magnitude larger than by the two previously discussed mechanisms (atomic volume increases or gas in small voids).

The observed changes in core volume of the fuel plates provide an insight into the physical variations that occur during irradiation. Up to fission densities of about 7×10^{20} f/cc, there appears to be a minor increase in the plate volume in the UAl₃ composition No. 1 (cf. eg, Plate 1-584 was irradiated to about 7.3×10^{20} f/cc with a measured volume change of 0.6 percent). Between 7.3 and 16.3×10^{20} f/cc, a more significant increase occurs (cf. eg, Plate 1-587 irradiated to 16.3×10^{20} f/cc with a measured volume change of 5.1 percent). Based on atomic volume [using Equation (1) above], the change in core volume was calculated to be 11.8 percent for Plate 1-587. It appears, therefore, that the UAl₃-aluminum fuel has the inherent ability to accommodate 6.7 percent growth within the structure of the fuel core. Voids created during fabrication could accommodate 4.6 percent growth as computed from the following relationship:

$$\frac{\Delta V}{V} (\text{fab}) = \frac{V_{\text{pre, obs}} - \left(\frac{W_1}{\rho_1} + \frac{W_2}{\rho_2} + \dots + \frac{W_n}{\rho_n} \right)}{V_{\text{core}}} \times 100 \quad (3)$$

where

W = the weight of a given component in the core

ρ = the density of a given component in the core

and the other symbols are defined above.

Voids produced in the core as a result of the reaction of the intermetallic compound with the aluminum matrix could account for about 0.9 percent volume percent. The pertinent reaction is

The ratio of the product volume to reactant volume is 0.976. The change in volume occurring from this reaction was computed from

$$\frac{\Delta V}{V} (R) = \frac{\left(V_{\text{UAl}_3} + V_{\text{Al combined}} \right)}{V_{\text{core, pre}}} \times \left(1 - \frac{V_{\text{products}}}{V_{\text{reactants}}} \right) \times 100 \quad (4)$$

A third mechanism, the fission product occupation of interstitial or substitutional sites within the "defect" UAl_4 lattice, apparently accommodates some growth. This is particularly evident in view of the fact that 3 to 4 percent voids remain in the core matrix after irradiation, thus indicating that most of the 4.6 percent fabrication voids had not been utilized to accommodate the fission products.

The observed swelling is much less than would be expected from creep or yield phenomena and is less than would be predicted from the formation of a regular array of vacancy-induced gas bubbles (0.1 to 1 micron). A tabulation of these results and those of Plate P-1-584 is in Table XIV.

TABLE XIV

FISSION PRODUCT ACCOMMODATION WITHIN
THE CORES OF THE THREE TYPE FUEL PLATES

Plate Number	Dispersed Material	Irradiation Exposure 10^{20} fiss/cc	Observed Percent $\frac{\Delta V}{V}$ (obs)	Atomic Volume Percent $\frac{\Delta V}{V}$ (exp)	Percent Accommodated In-Core	Fabrication Voids Percent $\frac{\Delta V}{V}$ (fab)	Reaction Voids Percent $\frac{\Delta V}{V}$ (R)	Microstructure Estimate of Percent Voids After Irradiation
P-1-587	UAl ₃	16.3	5.1	11.8	6.7	4.6	0.9	3-4
P-1-584	UAl ₃	7.3	0.6	5.3	4.7	4.6	0.9	3-4
P-2-598	U ₃ O ₈	16.2	7.3	11.5	4.2	5.2	6.4	7-9
P-2-599	U ₃ O ₈	6.3	0.2	4.6	4.4	4.5	6.9	7-9

Plate Number	Dispersed Material	Irradiation Exposure 10^{20} fiss/cc	Observed Percent $\frac{\Delta V}{V}$ (obs)	Atomic Volume Percent $\frac{\Delta V}{V}$ (exp)	Particle Diameter Percent $\frac{\Delta V}{V}$	Original Percent Voids Within Nb Casing	Percent Measured Voids After Irradiation	Percent Voids Increase During Irradiation	Percent $\frac{\Delta V}{V}$ (fiss gas) Equation (2)
O-4-844	Nb-coated UO ₂	17.0	11.8	12.0	7.3	1.6	4.6	3.0	2.8

[a] Reference 12 shows that the crystallographic arrangement of atoms in this compound should result in UAl_4 but that a "defect" lattice exists in which fewer than the stoichiometric number of uranium atoms reside with a resultant ratio more nearly that of UAl_5 .

A comparison of the pre- and post-irradiation microstructures, Figure 19, shows voids are not being produced in the UAl_3 fuel particles. Furthermore, it appears that the fabrication-induced particle cracking has disappeared.

The voids in the microstructures of the U_3O_8 plates, shown in Figure 20, suggest that gross swelling has occurred in the plate. The many voids of various sizes in the fuel particles range in size up to ≈ 60 microns and produce a froth-like appearance in the U_3O_8 -Al reaction product. Since the voids are much larger than ≈ 1 micron (the approximate size for surface tension control), creep or yielding is indicated. However, the observed core volume changes show that the gross swelling mechanisms were not operative. The gases, then, are not causing creep or yielding but are filling voids produced during the U_3O_8 -Al reaction (or voids remaining from fabrication). If Plate P-2-598 (U_3O_8) is analyzed, for example, the observed percent $\frac{\Delta V(\text{obs})}{V}$ is only 7.3 percent compared to an expected volume change of 11.5 percent. The growth accommodated in the core is 4.2 percent. Voids created in the core during fabrication were 5.2 percent [by Equation (3)], and those produced according to the reaction $[3\text{U}_3\text{O}_8 + 61 \text{ Al} \rightarrow 9\text{UAl}_5 + 8\text{Al}_2\text{O}_3]$ ($\frac{\text{volume products}}{\text{volume reactants}} = 0.871$) are 6.4 volume percent of the core, by Equation (4). It is not surprising to see voids in the core, therefore, because there should either be 7.4 percent voids ($6.4 + 5.2 - 4.2$) or a collapse of the plate by that amount. Such a collapse, of course, would be reflected by a negative percent $\frac{\Delta V(\text{obs})}{V}$. The results for Plates P-2-598 and -599 are in Table XIV.

The niobium-coated UO_2 plate cores more nearly approach the anticipated swelling. As an example, Plate 0-4-844 had an observed volume change of 11.8 percent compared to an expected 12.0 percent. At first appraisal, this would indicate that the UO_2 - ZrO_2 particles had very little capacity to accommodate fission products. Examination of the microstructures revealed matrix cracks developing and particle cracking voids enlarging with increased irradiation which must be accounted for in the observed swelling. Based on the measured increase in particle diameters, 7.3 percent swelling was produced within the Nb casings. The remaining swelling ($11.8 - 7.3$ or 4.5 percent) then was produced outside the Nb shells primarily by a growth interaction of the agglomerated particles on one another.

The reported total voids in the core after fabrication, but before irradiation, were 1.6 volume percent, and these were present inside the Nb casings. After irradiation, an estimate from the microstructure indicated the void volume increased to 4.6 percent of the core (see Figure 21). There was 3.0 volume percent voids produced during irradiation, and these ranged from 1 to 5 microns in size. From Equation (2), the possible fission-gas-induced void-volume change is 2.8 percent and is nearly equal to the measured 3.0 percent increase.

Comparing the three high burnup samples on a swelling basis [11.8 percent for the UO_2 (possibly 7.3 in proper dispersion), 7.3 percent for the U_3O_8 , and 5.1 percent for the UAl_3], there appears to be no benefit from the Nb coatings.

1.3 Blistering Mechanisms

It is concluded from the foregoing discussion that the gas pressure in the voids (even in the 60-micron U_3O_8 voids) was not sufficient to cause

AS POLISHED

50X

AS POLISHED

500X

BEFORE

Fig. 19 (Part I) Microstructures of UAl₃ cores before irradiation.

AS POLISHED

50X

AS POLISHED

500X

AFTER

Fig. 19 (Part II) Microstructures of UA_{13} cores after irradiation. Fabrication-induced particle cracking has disappeared (Plate P-1-587).

AS POLISHED

50X

AS POLISHED

500X

BEFORE

Fig. 20 (Part I) Microstructures of U_3O_8 cores before irradiation.

AS POLISHED

50X

AS POLISHED

500X

AFTER

Fig. 20 (Part II) Microstructures of U_3O_8 cores after irradiation. The U_3O_8 reacts with the aluminum matrix producing voids which are filled with fission gas giving a froth-like appearance to the fuel particles (Plate P-2-598).

AS POLISHED

500X

COATED UO_2 IRRADIATED TO 14×10^{20} FISS/CC
PLATE 0-4-848

AS POLISHED

500X

COATED UO_2 IRRADIATED TO 17×10^{20} FISS/CC
PLATE 0-4-844

Fig. 21 Microstructures of Niobium-coated UO_2 fuel cores after irradiation. The volume of voids in the particles is a function of radiation exposure. Plate 0-4-844 has an estimated 17 percent voids within a given particle amounting to 4.6 void volume percent in the core.

creep or yielding in the plates at the conditions of time, temperature, loop pressure, and irradiation exposures experienced. Gas pressure which is insufficient to cause creep in small voids, however, can cause blistering if there is a mechanism of void interconnection. The minimum radius of interconnecting which is necessary to cause blistering can be calculated. From the perfect gas law, the maximum pressure (P) which could exist in interconnected voids is

$$P = \frac{nRT}{V} \quad (5)$$

where

V is the void volume in the core after irradiation

n is the total moles of fission-produced gas (including He)

R is the universal gas constant

and

T is the absolute temperature.

The formula for the maximum stress of a circular plate with fixed edges and uniform loading is

$$(\sigma)_{\max} = \frac{3}{4} \frac{qa^2}{h^2} \quad (6)$$

Solving this equation for "a", the minimum radius of interconnecting is

$$a = \sqrt{\frac{4h^2\sigma}{3P}} \quad (7)$$

where

h is the cladding thickness corrected for corrosion

σ is the creep limit at operating temperature

and

P is the pressure calculated from Equation (5).

For an extreme case, consider the U_3O_8 plate with the maximum exposure of 16.2×10^{20} at a temperature of $500^\circ F$ with a cladding thickness, h, of 0.012 inch and a 1000-hr creep limit of 1200 psi. The minimum radius of interconnecting is 0.00605 inch. The pressure in the voids from Equation (5) is 6290 psia. If the loop pressure, 475 psia, is subtracted to give a differential pressure of 5815 psi, the minimum radius calculated is slightly larger or 0.00644 inch.

If the largest void in the U_3O_8 plate is considered to be 0.0024 inch and this is compared with the minimum diameter for interconnecting (0.0121 inch), there is a factor of about 5.0.

In the current irradiation test, the Nb-coated UO_2 particle plates exhibited a mechanism of interconnecting.

As expected, core growth is restrained in the length and width dimensions of the plate, but proceeds in the thickness direction (see Section III-1.6). The portions of matrix indicated by the arrows (Figure 22) are then in tension and

Fig. 22 Microstructures of Plate 0-4-852, Nb-coated UO_2 , after irradiation to 11.3×10^{20} f/cc. The arrow indicates areas of matrix cracking which caused interconnecting.

have been stressed to failure. When a portion of this plate was raised to a temperature only slightly higher than reactor conditions (540°F), blistering occurred (see Figure 23).

In previous irradiation tests^[7], it was recognized that the U_3O_8 -aluminum fuel dispersions also have a potential mechanism of interconnecting. The steps to failure which were recognized at that time are as follows:

- (1) The particles interconnect due to a reaction between U_3O_8 and aluminum. During this reaction, large amounts of aluminum diffuse from the matrix into the particle thus causing the particle to grow and the matrix to diminish.
- (2) Separation occurs at the particle-matrix interface. Since the particles are grossly interconnected, the particle-matrix interface is a continuous network.

Fig. 23 Photograph of Nb-coated UO_2 plate after annealing at 540°F (Plate 0-4-852).

- (3) When interconnecting spreads to a critical diameter, as calculated by Equation (7), the total force of the gas pressure is sufficient to cause blistering. In actuality, the critical diameter is expected to be larger than that calculated in Equation (7) because all the fission gas produced is not released to the voids. Blister diameters produced in-pile were ≈ 0.5 inch in diameter.

Figure 24 shows the particle interconnecting and the particle-matrix separation which occurred during this previous in-pile test. The matrix had been reduced from 79 to 35 volume percent of the core. It is clear that after incompatibility has promoted particle interconnecting, the resistance to blistering depends on the bond at the particle-matrix interface.

The U_3O_8 , G-12, Cycle 68 and 69 test plates being reported herein did not blister in-pile as might be expected from this previous experience. Plate P-2-593 resisted blistering in a post-irradiation anneal test up to 750°F (see Figure 25).

However, at a particular stage during the irradiation, there was particle-matrix separation (Figure 26) in localized areas. There was also abundant evidence of particle interconnecting (Figure 27). It can be said that under the sample irradiation conditions of the G-12 loop for Cycles 68 and 69, gross particle-matrix separation did not occur and, therefore, in-pile blistering of the U_3O_8 plates did not develop.

Fig. 24 Microstructure of U_3O_8 -aluminum plate which was irradiated to 10×10^{20} f/cc. Interconnecting of the particles was followed by interconnecting of particle-matrix interface voids (E-28).

Fig. 25 Plate P-2-593, 41 wt% U_3O_8 , after the 750°F anneal.

Fig. 26 Microstructure of U_3O_8 Plate P-2-599 irradiated to 6.3×10^{20} f/cc. Note the area of particle-matrix separation.

As a further check on the blister resistance of this set of plates, a post-irradiation anneal test was run on the UO_2 and UAl_3 as well as on the U_3O_8 . The results in Figure 28 show the changes in core densities caused by 30-min incremental heat treatments. The UO_2 plate blistered at the first temperature increment as a result of the matrix cracking caused by irradiation swelling. The U_3O_8 plates resisted blistering until the aluminum cladding had lost the major part of its strength (as a result of elevating the temperature) thus indicating that gross particle-matrix separation had not occurred on this set of plates. The UAl_3 plates showed the greatest resistance to blistering.

1.4 Superior Performance of UAl_3

Again, as in previous irradiation tests^[7], the UAl_3 composition has shown superior performance. The large capacity of the core to retain growth from fission atomic volume increase prevents the matrix-cracking method of interconnecting as seen in the coated UO_2 plates.

The fact that there is only 1/3 the amount of aluminum diffusion from the matrix into the particle, as in the case of the U_3O_8 , is assurance that particle interconnecting will not occur (Figure 29). During this test series and the previous series, there has not been an in-pile failure of UAl_3 or any observed mechanism of void interconnection. The lowest temperature value for blistering in post-irradiation anneal tests obtained from these high-exposure test plates or those of the Reactor Fuels and Materials Program^[13] was 850°F (Figure 30).

UNIRRADIATED: THE PARTICLES ARE DISTINCTLY SEPARATE.

50X

IRRADIATED TO 17×10^{20} ; GROSS PARTICLES INTERCONNECTING (PLATE NO. P-2-598).

50X

IRRADIATED TO 8.4×10^{20} ; SOME PARTICLES INTERCONNECTING (PLATE NO. P-2-614).

50X

Fig. 27 Microstructures of U_3O_8 plates irradiated to various levels. The particles interconnect due to a reaction between U_3O_8 and Al.

Fig. 28 Post-irradiation annealing results.

AS POLISHED

50X

FUEL PARTICLES HAVE REMAINED DISTINCTLY
SEPARATE AND ARE SURROUNDED BY MATRIX.

PLATE P-1-587-UAl₃

AS POLISHED

50X

FUEL PARTICLES SHOW GROSS INTERCONNECTION.

PLATE P-2-598-U₃O₈

Fig. 29 Microstructure of UAl₃ compared to U₃O₈ which has been irradiated to 16×10^{20} under similar conditions. Interconnecting does not occur in the UAl₃.

≈ 1X

Fig. 30 Photograph of Plate P-1-588, 51 wt% UAl_3 , after annealing at 850°F.

At these temperatures, aluminum has very little strength, and failure is by gross yielding of the matrix rather than clad buckling resulting from interconnecting.

A comparison of the three materials (Figure 31) shows that fission gas bubbles are being generated within the ceramic fuel particles (UO_2 and U_3O_8) but not in the uranium-aluminum intermetallic fuel particles. In a summary of fission-gas behavior[14], Morrison has made reference to studies made on ceramic (UO_2) pellets[15, 16]. It was found, in the temperature range of interest, that fission gases were released from the surface of the sample at a rate dependent on λ (the decay constant of a particular isotope).

On the other hand, Reynolds, who studied the uranium-aluminum metallic system[17], found there was no measurable loss of fission gas during annealing periods up to three weeks and for temperatures up to 1180°F (640°C), the transformation temperature. It is expected that blister anneal temperatures would approach this value if helium were eliminated from the system.

1.5 Mechanical Properties

The fission and radiation effect on mechanical properties which might be expected in the fuel materials is an increase in strength with an accompanying

 $UA1_3$

P-1-587

500X

 U_3O_8

P-2-258

500X

Nb-coated UO_2

O-4-844

500X

Fig. 31 Microstructures of the three fuel materials irradiated to the highest burnup. Gas bubbles were being generated within ceramic fuel particles (UO_2 and U_3O_8) but not in the intermetallic fuel particles ($UA1_3$).

loss of ductility. A bend test was devised to determine the relative extent of damage in the radiation-embrittled fuel plates should the plates be required to deflect during reactor operation. The degree and radius of bend were selected to be more severe than that which might be expected in an actual element but were slight enough to capture the effects of progressive radiation exposure at the test levels. Based on previous experience and engineering judgment, an angle of 5 deg, permanent set, over a 1-inch-diameter mandrel was selected and used for all samples tests. (A range of mandrel sizes and a range of bend angles could not be used because only small amounts of materials were available.) This would be equivalent to a 38-mil deflection in a 2-inch-wide flat plate.

Figure 32 shows the results of the tests which cover the range from UAl_3 at 7×10^{20} f/cc, which did not crack, to U_3O_8 at 16×10^{20} f/cc, which showed cracking in the particles and matrix. The intermediate exposures (and U_3O_8 at 6×10^{20} f/cc) showed particle cracking arrested at the matrix. All cracking was transverse and confined to the core and the apex of the bend.

If the worst case is considered to be the high-burnup U_3O_8 sample with all the fission gas in voids, giving a pressure of 6290 psi (from Section III-1.3 of this report) on a transverse crack completely across the 20-mil core, the stress in the cladding would be 4200 psi and is about equal to the 500°F yield strength or 4000 psi. Embrittlement is not considered important for the following reasons:

- (1) Cracking with exposures up to and including 12×10^{20} f/cc was confined to particles
- (2) Total fission gas was not released to the voids
- (3) The severity of the bend test is greater than expected in a plate of an element.

The room temperature hardness of the reacted U_3O_8 particles is greater than that of the UAl_3 particles thus indicating more embrittlement and accounting for the U_3O_8 particles cracking at lower exposure levels than the UAl_3 , as shown in Figure 32. The high-exposure U_3O_8 showed a drop in particle hardness, but this is attributed to the high degree of porosity which had developed.

A fission hardening effect was detected in the UAl_3 plate matrix (Figure 33) and to a lesser extent in the U_3O_8 plate matrix. The coated UO_2 plate matrix showed only the amount of hardening which might be expected from radiation damage. A trend of hardening or softening could not be established for the 6061-0 cladding material which varied from dpn 35 to dpn 60 and which has considerable variation in that range before irradiation.

The hardening of the matrix, which reached dpn 110 for some UAl_3 plate matrices, is considered beneficial as long as the matrix has sufficient ductility to resist brittle fracture or to arrest cracks which may form in the particle, as indicated previously in Figure 32, when the required deformation is imposed.

1.6 Dimensional Changes

During the corrosion of the aluminum cladding, weight change data show there may be a gain or loss. Oxygen combines chemically with the aluminum to increase

$UAl_3 + Al$

3×10^{20} FISS/CC
NO CRACKING
PARTICLE = 320 DPN, MATRIX = 90 DPN
P-1-584

$U_3O_8 + Al$

6×10^{20} FISS/CC
PARTICLE CRACKING
PARTICLE = 420 DPN, MATRIX = 70 DPN
P-2-599

9×10^{20} FISS/CC
PARTICLE CRACKING
PARTICLE = 280 DPN, MATRIX = 110 DPN
P-5-622

8×10^{20} FISS/CC
PARTICLE CRACKING
PARTICLE = 505 DPN, MATRIX = 60 DPN
P-2-614

12×10^{20} FISS/CC
PARTICLE CRACKING
PARTICLE = 340 DPN, MATRIX = 105 DPN
P-5-623

16×10^{20} FISS/CC
PARTICLE CRACKING AND MATRIX
CRACKING
PARTICLE = 295 DPN (VOID EFFECT)
MATRIX = 85 DPN
P-2-598

Fig. 32 Microstructures of 5-degree bend test specimens comparing the transverse cracking observed in the UAl_3 composition to that in the U_3O_8 composition for various radiation exposures.

Fig. 33 Matrix hardness as a function of fission density.

thickness, while some of the aluminum may go into solution or the corrosion product sloughs off to decrease thickness. Because of this, small dimensional changes, approaching the corrosion loss, cannot be interpreted as swelling or densification of the fuel plates.

The dimensional data do show that significant volume changes do not come about as length and width changes and, therefore, appear as thickness changes. In Table XV, representative lengths and widths of the various compositions are

TABLE XV
LENGTHS AND WIDTHS OF VARIOUS SAMPLE
FUEL PLATES BEFORE AND AFTER IRRADIATION

Plate Number	Composition Number	Cycles Irradiated	Length Before (in.)	Length After (in.)	Width Before (in.)	Width After (in.)
584	1	68	5.750	5.748	1.250	1.251
587	1	68 and 69	5.750	5.749	1.250	1.253
598	2	68 and 69	5.749	5.746	1.251	1.250
844	4	68 and 69	5.752	5.750	1.251	1.259
599	2	68	5.751	5.747	1.251	1.250
812	3	68	5.750	5.748	1.250	1.250

shown which all fall within the expected corrosion fluctuation except Composition 4, the coated UO_2 , which shows slight width increases.

Plate 844, for example, has a core volume change of 11.8 percent based on density data. In terms of plate length increases, this would be equal to 0.64 inch or in width increases, would be equal to 0.12 inch; and in terms of plate thickness increases, it would only be 0.0024 inch (approaching corrosion change). The width of this particular plate increased 0.008 inch during irradiation thus indicating that 6 percent of the swelling was expressed as a width change and 94 percent was expressed as thickness change (the length remained essentially the same).

1.7 Metallography

Microstructures have been used extensively in the previous sections of this report for illustration. This section will serve to make additional comparisons. Specifically, the effects of different irradiation conditions on the microstructures of the three fuels, verification of the eutectic bonding of the thermocouple plates, and coating metallography are covered.

The relative stability of the fuel materials has already been discussed, but the photomicrograph arrangement in Figures 34 and 35 shows, for ready

UO_2 U_3O_8 $UA1_3$

CONDITIONS

P-3-805

P-1-589

28 TO 31 PERCENT
BURNUP 2.5×10^{14} F/SEC-G
U-235

BELOW 230°F

O-4-843

P-2-599

P-1-584

24 TO 29 PERCENT
BURNUP 6.5×10^{14} F/SEC-G
U-235

325 TO 400°F

O-4-844

P-2-598

P-1-587

60 TO 65 PERCENT
BURNUP 7.5×10^{14} F/SEC-G
U-235

360 TO 520°F

Fig. 34 Microstructures (50X) of the three fuel types after exposure to various irradiation conditions.

Fig. 35 Microstructures (500X) of the three fuel types after exposure to various irradiation conditions.

comparison, the large microstructural changes in the oxide plates and the lack of change in intermetallic plates. With increased temperature and neutron flux, the UO_2 microstructure shows matrix cracking, particle growth, and increased fission-gas porosity. The U_3O_8 shows particle matrix reaction, particle interconnecting, loss of matrix, and large void formation. There are no detectable changes in the UAl_3 microstructures in these exposure ranges.

Each of the split fuel thermocouple plates, used in Reactor Cycle 69, was sectioned in the area of thermocouple insertion to check the silicon-aluminum eutectic bond between the inner 80-mil-thick solid aluminum plate and the outer 35-mil-thick fuel plates. In every case, the bond was sound. The interfaces were detectable in some samples as a solidified aluminum-silicon eutectic layer ≈ 0.1 mil thick (Figure 36).

Fig. 36 Microstructure showing typical bond in thermocouple plates (Plate P-2-677).

As a check on the Demitron (an eddy-current instrument used for measuring corrosion films), the aluminum oxide coating was measured metallographically on a number of the mounts from plates that were not electrolytically stripped. For comparison, metallographic mounts were selected from plates which gave nearly uniform Demitron readings. Each mount was scanned metallographically to find the maximum coating thickness (Figure 37) since the coatings are quite friable and could only be detected on 2 percent of the surface after polishing.

Comparing the maximum metallographic readings of a particular mount in the Demitron readings from that region (Table XVI), the greatest deviation is found to be 0.42 mil; the average deviation is 0.21 mil. The Demitron values,

Fig. 37 Typical microstructure of aluminum oxide coating used for coating thickness measurements (Plate P-1-589).

TABLE XVI

METALLOGRAPHY MEASUREMENTS VERSUS DEMITRON MEASUREMENTS

<u>Mount Number</u>	<u>Plate Number</u>	<u>Maximum Thickness Metallography (mils)</u>	<u>Demitron Thickness (mils)</u>	<u>Difference</u>
264	P-5-622	0.89	0.60	+0.28
215	0-3-815	1.28	0.88	+0.40
215	0-3-815	1.30	0.88	+0.42
216	0-3-815	0.75	0.88	-0.13
209	0-3-806	1.16	0.82	+0.34
210	0-3-806	1.09	0.82	+0.27
210	0-3-806	0.89	0.82	+0.07
270	P-5-623	0.73	0.58	+0.15
197	0-3-803	1.10	0.75	+0.35
198	0-3-803	0.85	0.75	+0.10
197	0-3-803	1.04	0.75	+0.29
203	0-3-805	0.42	0.30	+0.12
204	0-3-805	0.38	0.30	+0.08

which follow closely the predicted rate of oxide film buildup, were correlated with thermocouple temperature data; this is discussed in Section III-2.

1.8 Fuel Burnup

In calculating the fuel burnup, either of two assumptions can be made. It may be assumed that the thermal neutron flux remains constant such that the burnup is calculated from the following relationship:

$$\frac{N}{N_0} = e^{-\sigma_T \Phi \Delta t}$$

where

N is the number of fissile atoms remaining in a unit volume after the time increment, Δt (in seconds)

N_0 is the original number of fissile atoms in a unit volume

σ_T is the total microscopic cross section in cm^2

and

Φ is the thermal neutron flux in $\text{n/cm}^2 - \text{sec}$.

Or it may be assumed that the fission rate ($\frac{dN}{dt}$) is a constant. In this case, $N_0 - N = N_0 \sigma_f \Phi \Delta t$ for fissioning, and the total burnup is calculated from $\frac{\sigma_T}{\sigma_f}$ ($\frac{N_0 - N}{N_0}$) by assuming the absorption process follows directly the fission process.

In the results that follow, a fission cross section and a total cross section of 580 and 680 barns, respectively, were used.

The total power generated in the G-12 fuel sample assembly was computed periodically from the measured increase in the bulk coolant temperature and loop coolant flow. Two corrections to these values were required in order to ascertain the fission power of the fueled samples. The power generated by gamma absorption in the sample support assembly was calculated assuming an average gamma heat rate of 25 watts per gram.

The second correction arose from the loss of heat through the piping below the reactor core to the ETR primary coolant. The fission power generated in the fueled samples, expressed in millions of Btu/hr, is correlated with irradiation time in hours at full reactor power in Figure 38. It can be seen that the fission power is constant within about ± 10 percent for all of Cycle 68 and for the first 200 hours of Cycle 69.

Fig. 38 Sample heat generation as a function of ETR operating time -- Cycles 68 and 69.

It was assumed in computing the fission density or burnup that the fission rate was a constant during Cycle 68 and was 93 percent of the values measured in the ETRC.

The method used to calculate the fission density obtained in Cycle 69 consisted of assuming a constant fission rate of 89 percent of that measured in ETRC for Cycle 68 for the first 200 hours of Cycle 69 and of performing a numerical integration assuming a constant fission rate equivalent to the measured average for 50-hour increments beyond the 200-hour point. The fission rates were measured in the ETRC for Cycle 69 in a simulated loading; the results were found to be within ± 10 percent of those measured for Cycle 68; therefore, the Cycle 68 data were utilized. The results of these calculations are presented in Tables XVII and XVIII.

Tables XIX and XX compare the calculated fission densities with those measured at ICPP by isotopic burnup analyses for Cycles 68 and 69.

The correspondence obtained between the measured and calculated fission densities is considered acceptable for those plates which were irradiated in Tiers 3, 4, or 5 during either Cycle 68 or 69 and which were not niobium-coated spherical UO_2 . For the former, the ratio of the isotopic analysis to the calculated varied from 0.93 to 1.07. For those plates in Tiers 3, 4, or 5 which were irradiated during both cycles and which did not contain niobium-coated UO_2 , the ratio varied from 0.87 to 0.94. Most emphasis is placed upon the comparison for the indicated fuel plates since the samples which were analyzed chemically consisted of one-quarter of the plate taken below the center line and the axial fission rate profiles are more nearly constant over this length for these three tiers. The ratio is much less than 1.0 (0.55 and 0.74) for the two niobium-coated spherical UO_2 plates; the niobium layer with an absorption cross section of 1.1 barns may be the reason for this discrepancy.

TABLE XVII

CYCLE 68 CALCULATED FISSION DENSITIES

<u>Plate</u>	<u>Plate Number</u>	<u>Average Fission Rate^[a] 10¹⁴ f/sec-g</u>	<u>Average Fission Density 10²⁰ f/cc</u>
<u>Tier 1</u>			
1	P-1-651	3.5	3.6
2	P-1-589	2.9	3.0
3	P-2-614	2.9	3.0
4	O-3-805	3.1	3.2
<u>Tier 2</u>			
1	P-5-659	7.1	7.4
2	P-5-621	5.8	6.0
3	P-5-625	5.8	6.0
4	P-5-623	6.2	6.4
<u>Tier 3</u>			
1	P-1-654	8.9	9.3
2	P-1-584	7.3	7.6
3	P-2-593	7.3	7.6
4	O-4-843	7.8	8.1
<u>Tier 4</u>			
1	P-2-658	9.7	10.1
2	P-2-598	8.0	8.3
3	P-1-587	8.0	8.3
4	O-4-844	8.5	8.8
<u>Tier 5</u>			
1	P-1-653	8.3	8.6
2	O-4-848	6.8	7.1
3	P-2-599	6.8	7.1
4	P-1-588	7.3	7.6
<u>Tier 6</u>			
1	O-3-827	5.4	5.6
2	O-3-815	4.4	4.6
3	O-3-812	4.4	---
4	O-3-806	4.7	4.9

[a] 0.93 multiplied by ETRC measured values.

TABLE XVIII

CYCLE 69 CALCULATED FISSION DENSITIES

<u>Plate</u>	<u>Plate Number</u>	<u>Initial Average Fission Rate^[a] 10¹⁴ f/sec-g</u>	<u>Average Fission Density 10²⁰ fission/cc</u>
<u>Tier 1</u>			
1	P-1-674	3.4	4.6
2	P-1-589	2.8	6.8
3	P-2-614	2.8	6.8
4	O-3-805	3.0	7.4
<u>Tier 2</u>			
1	P-5-662	6.8	9.5
2	P-5-621	5.6	13.7
3	P-5-623	5.6	13.9
4	P-5-622	5.9	8.1
<u>Tier 3</u>			
1	P-1-676	8.5	11.8
2	P-1-585	7.0	9.6
3	P-2-601	7.0	9.6
4	O-4-852	7.5	10.4
<u>Tier 4</u>			
1	P-2-677	9.3	12.8
2	P-2-598	7.7	18.9
3	P-1-587	7.7	18.9
4	O-4-844	8.1	20.0
<u>Tier 5</u>			
1	P-1-673	7.9	11.1
2	O-4-848	6.5	16.2
3	P-2-593	6.5	16.7
4	P-1-588	7.0	17.2
<u>Tier 6</u>			
1	O-4-823	5.2	7.2
2	O-3-803	4.2	5.8
3	O-3-815	4.2	10.4
4	O-3-806	4.5	11.1

[a] 0.89 multiplied by Cycle 68 ETRC measurements.

TABLE XIX

COMPARISON OF MEASURED AND
CALCULATED FISSION DENSITIES FOR CYCLE 68

Plate Number	Irradiation Location (tier/plate)	Average Isotopic Fission Density 10^{20} fiss/cc	Cs-137 Fission Density 10^{20} fiss/cc	Calculated Average Fission Density ^[a] 10^{20} fiss/cc	Ratio of Isotopic to Calculated
P-2-599	5/3	6.6	5.9	7.1	0.93
P-5-625	2/3	5.1	5.0	6.0	0.85
P-1-584	3/2	7.4	7.0	7.6	0.97
0-3-812	6/3	3.9	4.2	3.9	1.0

[a] Corrected for sampling location.

TABLE XX

COMPARISON OF MEASURED AND
CALCULATED FISSION DENSITIES AT COMPLETION OF CYCLE 69

Plate Number	Irradiation Location (tier/plate)	ETR Cycles Operated	Measured Isotopic Fission Density 10^{20} fiss/cc	Calculated Average Fission Density 10^{20} fiss/cc	Ratio of Isotopic Result to Calculated
P-1-589	1/2	68 and 69	8.3	7.9	1.05
P-2-614	1/3	68 and 69	8.4	7.9	1.06
0-3-805	1/4	68 and 69	7.5	8.6	0.87
P-5-621	2/2	68 and 69	12.6	14.8	0.85
P-5-623	2/3	68 and 69	12.1	15.2	0.80
P-5-622	2/4	69	8.7	8.7	1.00
P-1-585	3/2	69	10.7	10.0	1.07
P-2-593	3/3 and 5/3	68 and 69	14.7	16.4	0.90
P-2-601	3/3	69	10.7	10.0	1.07
P-2-598	4/2	68 and 69	16.2	18.7	0.87
P-1-587	4/3	68 and 69	16.3	18.7	0.87
0-4-844	4/4	68 and 69	14.7	19.8	0.74
0-4-848	5/2	68 and 69	8.3	15.2	0.55
P-1-588	5/4	68 and 69	15.2	16.2	0.94
0-3-803	6/2	69	6.7	4.9	1.37
0-3-815	6/3	69	8.6	8.7	0.99
0-3-806	6/4	68 and 69	11.2	9.3	1.20

2. CLADDING CORROSION

2.1 General

Corrosion of aluminum alloys which might be used as the fuel core cladding in either ATR or HFIR was studied in ex-reactor experiments at Oak Ridge National Laboratory and reported in Reference 1. A correlation was developed for X8001 and 6061 aluminum alloys for surface heat fluxes greater than about one million Btu/hr-ft² when demineralized water, acidified with nitric acid, was passed at velocities in excess of 25 fps past the electrically heated specimens. For pH 5.0 ± 0.2, the correlation is

$$\chi = 443 \theta^{0.778} / e^{\frac{8280}{R}}$$

where

χ is the alumina corrosion film thickness in mils

R is the surface temperature (ie, coolant-oxide interface) in °Rankine
and

θ is the exposure time in hours.

For pH between 5.7 and 7.0, the coefficient is increased by a factor of 2.7.

The thermal conductivity of the alumina formed was determined in these ex-reactor studies to be 1.3 ± 0.2 Btu/hr-ft°F (Reference 1).

To calculate the oxide thickness in those cases where the surface temperature varied as a function of time, the following procedure was used:

- (1) The operating period was divided into time increments (usually 50 hours)
- (2) The oxide thickness in the first time increment was calculated from the above correlation assuming the surface temperature remained constant at its initial value
- (3) The time to form the calculated oxide thickness formed in the first time increment with the surface temperature for the second increment was calculated and added to the second time increment
- (4) The oxide thickness at the end of the second time increment was calculated.
- (5) Steps 3, 4, and 5 were repeated for each time increment to the completion of the irradiation.

2.2 Measured and Calculated Temperatures

The instrumented plate temperatures were monitored continuously during the course of the irradiation to provide data from which an estimate of the in-pile performance could be made.

Figures 39 through 47 present the measured temperatures as a function of operating time for Cycles 68 and 69. The measurements for Cycle 68 were not

Fig. 39 Measured temperatures in Tier 2, Cycle 68.

Fig. 40 Measured temperatures in Tier 3, Cycle 68.

Fig. 41 Measured temperatures in Tier 4, Cycle 68.

Fig. 42 Measured temperatures in Tier 5, Cycle 68.

Fig. 43 Measured temperatures in Tier 6, Cycle 68.

Fig. 44 Measured temperatures in Tier 3, Cycle 69.

Fig. 45 Measured temperatures in Tier 4, Cycle 69.

Fig. 46 Measured temperatures in Tier 5, Cycle 69.

Fig. 47 Measured temperatures in Tier 6, Cycle 69.

considered satisfactory since it was not possible with the equipment available at that time to insert the hot junction a sufficient distance into the plate to avoid the large temperature gradients existent at the edges of the fuel core alloy, and the radiographs of the completed assembly indicated that only those hot junctions in Tiers 2 and 5 were apparently in contact with the aluminum alloy between the two fuel cores.

Tables XXI and XXII present the calculated temperatures at beginning-of-core life for Cycles 68 and 69, respectively. The Dittus-Boelter correlation for the heat transfer coefficient together with an inlet coolant temperature of 110°F, a coolant velocity of 46.5 fps, and the measured in-pile heat generation rates were used in arriving at these surface temperatures. The temperatures cited are the "maximum nominal"; this term implies that the cited temperatures do not include allowances for off-normal conditions, such as thinner coolant channels, fuel heterogeneity, local neutron flux perturbations, and minimum heat transfer coefficients. The cited temperatures are the maximums of those which are expected to be obtained.

When the hot junction is not in contact with the aluminum alloy, some heat is probably conducted up the sheath of the thermocouple resulting in an erroneous reading of the local temperature by an amount proportional to the size and

TABLE XXI
CYCLE 68 THERMAL CALCULATIONS

Plate	Beginning-of-Life				Peak Clad-Oxide Interface Temperature During Irradiation	
					Based on	Based on
	Maximum Nominal q/A 10 ⁶ Btu/hr-ft ²	Average q/A 10 ⁶ Btu/hr-ft ²	Maximum Nominal Surface Temperature (°F)	Average Surface Temperature (°F)	Maximum Nominal Beginning-of-Life Conditions (°F)	Average Nominal Beginning-of-Life Conditions (°F)
<u>Tier 1</u>						
1	1.12	0.82	221	190	---	---
2	0.92	0.68	203	180	---	---
3	0.92	0.68	203	180	---	---
4	0.98	0.73	209	185	---	---
<u>Tier 2</u>						
1	1.94	1.65	301	275	---	---
2	1.61	1.36	273	250	313	275
3	1.61	1.36	273	250	313	275
4	1.72	1.46	283	260	333	290
<u>Tier 3</u>						
1	2.31	2.03	340	315	---	---
2	1.91	1.68	307	285	384	335
3	1.91	1.68	307	285	384	335
4	2.04	1.80	319	300	417	365
<u>Tier 4</u>						
1	2.43	2.23	355	340	---	---
2	2.01	1.84	322	310	422	390
3	2.01	1.84	322	310	422	390
4	2.15	1.97	334	320	461	415
<u>Tier 5</u>						
1	2.21	1.90	343	320	---	---
2	1.83	1.57	314	295	396	350
3	1.83	1.57	314	295	396	350
4	1.96	1.68	324	300	425	360
<u>Tier 6</u>						
1	1.61	1.22	301	275	---	---
2	1.33	1.01	288	265	330	285
3	1.33	1.01	288	265	330	285
4	1.42	1.08	296	270	338	295

composition of the gap between the hot junction and the metal and also proportional to the temperature gradient along the sheath. Photomicrographs of sectioned regions near the hot junction for Plate P-1-676 showed an annulus of 1 to 5 mils in thickness around the thermocouple sheath (Figure 48).

Attempts to correct these deficiencies were successful generally in the fabrication of the instrumented assembly used during Cycle 69, and these data are considered reliable essentially.

The change in the measured temperature as a function of operating time can only be caused by two factors for constant fission rate, coolant velocity, channel geometry, and inlet coolant temperature. Both of these are related to the overall thermal resistance between the point of measurement and the local bulk coolant. Either the formation of an insulating coating on the fuel plate surface, without a concomitant effect upon the film heat transfer coefficient, or a decrease in the thermal conductivity of the fuel core alloy could separately or together cause the measured temperature to increase. The effects are separable on the basis that the correlation for the formation of the alumina coating and its thermal conductivity, cited in Reference 1, applies. The increase in the cladding temperature can then be calculated. The results of such calculations are compared

TABLE XXII
CYCLE 69 THERMAL CALCULATIONS

Plate	Beginning-of-Life				Peak Clad-Oxide Interface Temperature During Irradiation	
	Maximum Nominal q/A 10 ⁶ Btu/hr-ft ²	Average q/A 10 ⁶ Btu/hr-ft ²	Maximum Nominal Surface Temperature (°F)	Average Surface Temperature (°F)	Based on	Based on
					Maximum Nominal Beginning-of-Life Conditions (°F)	Average Nominal Beginning-of-Life Conditions (°F)
<u>Tier 1</u>						
1	1.07	0.79	216	190	---	---
2	0.88	0.65	199	175	---	---
3	0.88	0.65	199	175	---	---
4	0.95	0.70	205	180	---	---
<u>Tier 2</u>						
1	1.86	1.57	293	265	---	---
2	1.54	1.30	266	245	326	280
3	1.54	1.30	266	245	326	280
4	1.65	1.39	276	250	317	290
<u>Tier 3</u>						
1	2.21	1.95	330	310	---	---
2	1.83	1.61	297	280	361	320
3	1.83	1.61	297	280	361	320
4	1.95	1.72	310	290	394	345
<u>Tier 4</u>						
1	2.33	2.13	345	330	---	---
2	1.92	1.76	313	300	466	405
3	1.92	1.76	313	300	466	405
4	2.06	1.88	324	310	514	440
<u>Tier 5</u>						
1	2.12	1.82	333	310	---	---
2	1.75	1.50	305	285	430	355
3	1.75	1.50	305	285	430	355
4	1.88	1.60	315	295	468	385
<u>Tier 6</u>						
1	1.54	0.99	293	255	---	---
2	1.27	0.82	280	250	313	275
3	1.27	0.82	280	250	342	275
4	1.36	0.88	288	250	363	275

with the measurements in Figures 49 through 57. It was assumed in the calculations that the fission rate was constant during all of Cycle 68 and for the initial 200 hours of Cycle 69; these assumptions are substantiated by the data presented in Figure 38. The comparison for the initial 200-hour period of Cycle 69 is considered the most reliable that can be provided by the data obtained during the irradiations.

More detailed comparisons of the calculated temperature increases with the measurements for the initial 75 hours are presented in Figures 58 and 59. These data are presented since it appeared that the calculations consistently overestimate the increase in temperature for this period.

The calculated cladding temperatures as a function of operating time for the uninstrumented plates are shown in Figures 60 and 61.

In attempting to assess the uncertainty in the beginning-of-life surface temperatures cited in Tables XXI and XXII, either of three different means may be utilized. The temperatures cited were calculated on the basis of the measured heat generation rate in the experiment and the axial and radial fission rates as measured in the ETR Critical Facility. The sample heat generation rate is

Fig. 48 Photomicrograph of sectioned regions near the hot junction for Plate P-1-676.

Fig. 49 Measured and calculated temperature changes for Tier 2, Cycle 68.

Fig. 50 Measured and calculated temperature changes for Tier 3, Cycle 68.

Fig. 51 Measured and calculated temperature changes for Tier 4, Cycle 68.

Fig. 52 Measured and calculated temperature changes for Tier 5, Cycle 68.

Fig. 53 Measured temperature changes for Tier 6, Cycle 68.

Fig. 54 Measured and calculated temperature changes for Tier 6, Cycle 69. Calculated using 0.89 times Cycle 68 data.

Fig. 55 Measured and calculated temperature changes in Tier 3, Cycle 69.

Fig. 56 Measured and calculated temperature changes in Tier 4, Cycle 69.

Fig. 57 Measured and calculated temperature changes in Tier 5, Cycle 69.

Fig. 58 Tier 4, Cycle 69, temperature change as a function of operating time.

Fig. 59 Tier 5, Cycle 69, temperature change as a function of operating time.

Fig. 60 Calculated temperatures for Cycle 68 uninstrumented plates.

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Fig. 61 Calculated temperatures for Cycle 69 uninstrumented plates.

calculated from the increase in bulk water temperature and the coolant flow rate, and it is corrected for gamma heating of the in-pile assembly and for the heat loss to the ETR primary coolant in the piping below the core. Uncertainties in each of these variables may be assigned, and the overall effect upon the surface temperature may be evaluated.

The second method would be to calculate the temperatures at the locations of the thermocouples in the instrumented fuel plates and to compare these calculations with the measurements. In addition to the measured total sample heat generation and the axial and radial fission rates, it is necessary to have knowledge of the thermal conductivity of the 10-mil-thick fuel matrix. Measurements of the latter are not available currently. Further, the radial location of the hot junction and its contact with the metal center in the instrumented plate can affect the thermocouple reading significantly.

The third method includes all the possible variations that might possibly occur and are combined in the most adverse fashion similar in concept to that exercised when executing a "hot spot-hot channel analysis". The hot channel, heat flux, and hot spot factors developed during the hazard analysis for this experiment were 1.68, 1.27, and 1.35, respectively. For example, the application of these factors yields a variance of $\pm 150^{\circ}\text{F}$, for the hottest fuel plate (Tier 4, Plate 1). This variance represents two standard deviations from the nominal, since the individual factors are based upon two standard deviations. This approach is considered unduly pessimistic in estimating the uncertainty in beginning-of-life surface temperatures since none of the measured instrumented plate temperatures substantiated the wide range indicated. A comparison of the measured temperatures with the calculated temperatures at the beginning-of-life is presented in Table XXIII. It was assumed in the calculations that the thermal conductivity of the fuel matrix was the same as that of aluminum.

Perusal of the radiographs of the instrumented assembly revealed the following:

- (1) The thermocouples in Tier 4 were placed at nearly the same elevation and insertion distance from edge of fuel core alloy
- (2) The elevations of the thermocouples in Tier 5 were nearly the same, but the west thermocouple insertion distance was about 0.150 inch compared to 0.220 inch for the east junction
- (3) The west junction in Tier 3 was about 0.100 inch inserted; whereas, the east junction was about 0.200 inch inserted.

The conclusions derived from these observations were as follows:

- (1) The measured temperatures in Tier 4 should agree reasonably well with the calculations (for example, a decrease in the heat generation rate of 10 percent would decrease the calculated temperature to the lower measured temperature

TABLE XXIII

COMPARISON OF CALCULATED AND MEASURED
METAL TEMPERATURES FOR CYCLE 69 STARTUP

Tier	Calculated Temperature (°F)	Measured Temperature (°F)	
		East Thermocouple	West Thermocouple
2	319	287	304
3	361	328	295
4	377	365	350
5	362	353	333

(2) The west thermocouple readings in Tiers 3 and 5 should be less than the east thermocouple readings by an unknown amount

(3) The readings of the thermocouples in Tier 4 should be the most precise since the axial fission rate profile is essentially invariant in the region of the hot junction, and the calculated temperatures (see Tables XXI and XXII) should be no more than about 25°F higher than the actual using the measured metal temperatures as the criterion.

The uncertainties required to allow an evaluation of the beginning-of-life surface temperature variations and assigned values are the following:

Coolant temperature differential (1.02 for each resistance bulb)	1.04
Coolant flow rate	1.03
Gamma heating	1.10
Heat loss in loop piping	<u>1.00</u>
Total variation	1.18

For Plate 1, Tier 4 (the hottest plate), the resultant temperature uncertainty is 50°F; the value is less for all other fuel plates.

It is concluded that the beginning-of-life surface temperatures cited in Table XXI are probably within 25°F of the actual temperatures and that these temperatures are probably higher than the actual.

In consideration of the uncertainty of the maximum nominal clad-oxide interface temperature during the irradiation, two additional uncertainties are involved. They are as follows:

- (1) The rate at which the alumina coating forms
- (2) The thermal conductivity of the alumina coating.

The measurements made in the study reported here were insufficient to allow assignment of uncertainties to these two quantities. Hence, it is necessary to use ex-reactor data. Reference 1 states that for a constant surface temperature of 300°F and an exposure time of 400 hours, the alumina thickness at the 95 percent confidence level varied from 0.805 to 0.904 mil and, further, that the alumina thermal conductivity is 1.3 ± 0.2 Btu/hr-ft-°F at the same confidence level. These two variations at a surface heat flux of 2.0×10^6 Btu/hr-ft² could cause a variation of plus 28°F to minus 20°F in the cladding temperature. Combining these variations with those variations in surface temperature results in an estimate of the cladding temperature probably being within about 50°F of the reported cladding temperature.

2.3 Measured Alumina Thickness

The alumina thickness was measured in the Hot Cell at five locations along two axial lines about 1/4 inch from each edge on both sides of each of the irradiated fuel plates. The measurements were made with an eddy-current device, "Demitron Thickness Tester", Model D-2, with an 1/8-inch-diameter probe; the manufacturer states a precision of ± 10 percent of the thickness of coating being measured.

Figures 62 through 67 present the measured alumina thicknesses and the axial heat flux as a function of fuel plate length. The calculated oxide thicknesses are also shown for the maximum nominal calculated wall temperature for those plates where the heat flux at beginning-of-life was greater than one million Btu/hr-ft².

In general, it is observed that the measured oxide thickness correlates with the axial heat flux. The latter is essentially a representation of the axial wall temperature variation since the increase in bulk water temperature for a given tier is very small in comparison to the film temperature increase; hence, the wall temperature is directly proportional to the heat flux for a constant coolant velocity.

The calculated oxide thicknesses are greater than the measurements for the majority of the fuel plates. The most notable exceptions are the plates that operated in Position 4, Tiers 3 and 4, during Cycle 69. Subsequent to irradiation, it was found that the plate in Position 4, Tier 3, was severely warped which probably changed the local coolant velocities with a consequent increase in the surface temperatures and, therefore, increased oxide formation rates.

The appearance of the oxide formed in-pile did not conform with that obtained in the ex-reactor experiments. In the latter, it was reported that the oxide was essentially colorless; whereas, the oxide formed in-pile has from a light-tan to a dark-reddish-brown appearance. The results of a chemical analysis of a sample of the reddish-brown coating from Plate Number P-2-599 are presented in Table XXIV. If the coating were boehmite (aluminum oxide monohydrate), the weight percent Al would be expected to be 46 weight percent; whereas, 40 weight percent Al was obtained. In acquiring the coating sample for analysis, no attempt was made to strip the coating completely; but, rather,

Fig. 62 Tier-1, measured axial oxide thickness.

Fig. 63 Tier-2, measured axial oxide thickness.

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Fig. 64 Tier-3, measured alumina thicknesses.

Fig. 65 Tier-4, measured alumina thicknesses.

Fig. 66 Tier-5, measured alumina thicknesses.

Fig. 67 Tier-6, measured alumina thicknesses.

TABLE XXIV

CORROSION PRODUCT
CHEMICAL ANALYSIS

Element	Weight Percent
Aluminum	40.0
Iron	5.0
Magnesium	0.2
Silicon	0.1
Titanium	0.1
Lead	0.1
Chromium	0.2
Boron	0.02
Copper	0.03
Gallium	0.03
Manganese	0.05
Beryllium	0.002
Nickel	0.005
Zirconium	0.006

Major Crystalline Component:

Minor Crystalline Component:

the sample was obtained by scraping a small quantity from the surface. X-ray diffraction studies upon the same sample revealed the presence of $\alpha\text{-Fe}_2\text{O}_3$ with a small amount of $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$. If the iron oxide were formed prior to the alumina, the former would reside at the oxide-coolant interface, and the method used to acquire the sample would tend to overestimate the quantity of iron oxide present. The thickness of iron oxide for a given thickness of boehmite can be calculated from the results of the chemical analysis and the handbook values of the densities of the materials. It was calculated that 0.05 mil of iron oxide per mil of boehmite

was formed. Although this appears to be an insignificant quantity of iron oxide, it is conceivable that its presence could inhibit significantly the rate of boehmite formation.

The ORNL investigators reported a "new-loop effect"[1] in which the corrosion rate of 6061-0 aluminum was decreased in measurements conducted prior to pretreatment of the newly constructed stainless steel recirculating loop piping. A thin light-tan coating, reported as mostly iron, was found upon the aluminum oxide corrosion product. The loop was then pretreated by recirculating demineralized water at 545°F, which contained 500 ppm oxygen, for 96 hours. Subsequently, the measured alumina formation rate obtained in this loop agreed with measurements made in an older loop.

The supply coolant for the G-12 ETR Water Loop was obtained from the ETR primary coolant and flowed by the fuel specimens after an average residence time of about 30 seconds in the Type 347 stainless steel piping. The coolant was returned to the ETR reactor primary coolant discharge stream. Routine chemical analyses of the ETR primary coolant for iron showed no significant change during the loop operation from its normal value of about $1\mu\text{g}/\ell$.

All of the G-12 ETR Water Loop piping was pretreated at room temperature in a solution of the following composition prior to installation in the loop:

NaOH	3 pounds
Na ₂ HPO ₄	3 pounds
NaNO ₃	1 pound
H ₂ O	1000 pounds

Dousing in this solution was followed by a demineralized water rinse.

Following four cycles of operation, the G-12 loop was deactivated, and some of the piping was disassembled as a part of a loop revision. At that time, it was observed that there was a thin light-tan adherent coating on the inside of the piping. The results of chemical and metallurgical examinations of this film indicated it to be a very thin amorphous material. Stainless steel components immersed in the ETR primary coolant have been observed to be coated with a similar film.

The ex-reactor aluminum corrosion experiments showed a very sharp change in corrosion rate when the loop pH was increased from 5.0 ± 0.2 to 5.7. It was the intent, during the G-12 irradiation, to control the pH at 5.0 ± 0.2 ; this was accomplished according to the pH instrument which continuously measured and recorded the pH of a side stream of the loop effluent. Moreover, the behavior of the pH instrument was consistent throughout the irradiation since an increase in pH was obtained whenever the reactor power was decreased and vice versa. Measurements of the pH of loop effluent samples several days after the samples were taken showed the sample pH to be more variable than ± 0.2 . (See Section IV of this report.) These samples were found to contain air, and some change in the pH may have occurred during the storage period. Even though the G-12 pH instrument was carefully calibrated prior to the initiation of the experiment, nitric acid (pH of 2.0) was added at a rate of about 0.25 gph

while at full reactor power and the instrument was within the control limits, it is conceivable that the pH was not controlled within the 5.0 ± 0.2 range. The pH of the ETR primary coolant is measured with a portable pH meter at four-hour intervals whenever the coolant is flowing; these measurements are plotted as a function of full-power operating time in Figures 68 and 69 for Cycles 68 and 69, respectively. While the evidence may be controvertible, it

Fig. 68 Cycle 68 pH history.

Fig. 69 Cycle 69 pH history.

is reasonably clear that the limits of the pH were between 4.9 and 5.9 during the irradiation, with the most prevalent range of 5.0 to 5.5.

2.4 Discussion of Corrosion

The data obtained during these irradiations indicate general agreement with the correlation for the corrosion of 6061-0 or X8001 aluminum alloys developed from the ex-reactor experiments. This agreement may be coincidental, however, since the course of the corrosion phenomenon does not appear to be the same for both experimental programs. In-pile, the corrosion appeared to have a lower rate during a period, which was about 60 to 70 hours in duration, at the beginning of the irradiation. Following this induction period, the in-pile and ex-reactor corrosion rates were about the same up until about 200 hours at full power. Film spalling evidently occurred more readily in-pile since the calculated film thickness was roughly one-half that required in the ex-reactor work for spalling to begin.

One possible explanation of the behavior observed in-pile is that the ferrous and ferric ions in the coolant were reduced to elemental iron by the electrochemical differences of the aluminum and iron ions and the aluminum temperature. Following the deposition of a small amount of iron, this oxidation-reduction reaction was curtailed since the migration distance of the aluminum atoms was exceeded. Subsequently, the iron reacted with water and oxygen to form ferric oxide, and the aluminum reacted with oxygen and water to form alumina. The presence of iron oxide upon the plate surface may have inhibited the aluminum corrosion such that the substantial effect of pH that was shown in the ex-reactor experiments was not obtained in-pile.

The notion that surface temperature affects iron deposition may be argued on the basis of the in-canal observations wherein it was seen that the samples operating at the higher temperatures were evidently of a darker tone.

Other cations, anions, and complex ions in the ETR primary coolant may affect the aluminum corrosion rate in addition to the ferric or ferrous ions. Significant amounts of other ions were not found in the alumina layer, however, and, therefore, are not considered important.

The corrosion behavior of the 6061-0 aluminum cladding specified as the reference material for the ATR fuel plates cannot be predicted completely on the basis of the in-pile experiments reported herein. It is likely that the corrosion rate in the ATR will not exceed significantly that measured in the ex-reactor experiments since the in-pile results indicate some inhibiting effect of very small amounts of other ions. It is clear that the corrosion rate of the 6061-0 cladding should be measured in the ATR as a part of the post-neutron startup program, and these measurements are included in the present plans.

IV. LOOP COOLANT CHEMISTRY

Samples of the loop effluent were obtained periodically during the irradiation. Measurements of pH, conductivity, nitrate, nitrite, hydrogen peroxide, elemental hydrogen, oxygen, nitrogen, and argon and total gas concentration were made. The results are summarized in Table XXV. The results compare favorably with a similar analysis upon ETR primary coolant.

TABLE XXV

RESULTS OF LOOP EFFLUENT ANALYSES

Date	Cycle	pH	Conductivity (μ mhos/cm)	H ₂ O ₂ (ppm)	NO ₃ (ppm)	NO ₂ (ppm)	H ₂ (mol percent)	O ₂ (mol percent)	N ₂ (mol percent)	Ar (mol percent)	Total Gas (cc/liter)
12/2/64	68	5.2	4.1	12.0	0.6	0.05	7.5	22.5	69.2	0.9	$\approx$ 500[a]
12/3/64	68	4.8	10.0	12.0	0.5	0.05	9.8	21.0	68.4	0.7	$\approx$ 350[a]
12/8/64	68	5.2	2.6	13.0	1.0	0.05	1.7	14.2	45.7	38.3	$\approx$ 320[a]
1/26/65	69	5.9	2.3	2.0	0.5	0.02	----	----	----	----	24
2/3/65	69	5.0	1.9	10.1	0.2	0.03	50.6	26.1	22.8	0.5	25
2/5/65	69	4.9	1.4	8.2	0.2	0.02	16.1	22.6	60.5	0.8	$>$ 100[a]
2/8/65	69	5.9	1.5	9.4	0.2	0.02	50.5	26.9	22.1	0.5	23
2/10/65	69	5.9	1.6	8.3	0.5	0.01	51.3	25.6	22.6	0.5	26
2/12/65	69	5.6	1.0	9.7	0.1	0.01	47.1	27.2	23.1	0.5	22

[a] Air leaked into sample bomb.

V. CONCLUSIONS

The results of the experiments and evaluations reported herein permit the following conclusions regarding the reference fuel plate performance in ATR and HFIR:

- (1) Significant swelling of the reference fuel plates was not obtained at beginning-of-life surface temperatures and corresponding fission densities in the ranges anticipated during reactor operations
- (2) A possible mechanism by which U_3O_8 fuel plates could be weakened substantially was identified; a physical change in the matrix was evident (probably the result of the chemical reaction of U_3O_8 with aluminum), and it was observed that the voids were not retained within the fuel particles
- (3) The measured in-pile corrosion rate of 6061-0 aluminum agreed reasonably well with that measured in ex-reactor experiments.

The performance of the uranium aluminide in X8001 aluminum, clad with 6061-0, appeared to be superior to either the reference fuel material or UO_2 -aluminum cermet. It was found from studies of core volume changes as a function of fission density that the uranium aluminide cores accommodate the fission products more readily than the oxide-fueled samples. In addition, post-irradiation annealing studies indicated that equivalent or higher temperatures were required to obtain the same degree of swelling in comparison with the U_3O_8 fueled samples.

The performance of the niobium-coated spherical UO_2 - ZrO_2 fuel in X8001 aluminum matrix was found to be unsatisfactory for use in high-performance test reactors. It was observed that the particles did indeed retain the fission products but that the individual particles swelled to the degree that matrix cracking was obtained. Furthermore, post-irradiation annealing showed that matrix cracking permitted the fuel core to swell substantially at reactor operating temperatures.

One of the UO_2 fueled samples became severely warped during the irradiation. This behavior is attributed to poor distribution of the fuel within this plate.

VI. RECOMMENDATIONS

As a result of finding that the in-pile corrosion rate of 6061-0 agrees with that measured ex-reactor, it is evident that the fuel core temperatures to be encountered during reference operation of the ATR can be expected to be significantly greater than would be obtained if the adherent boehmite were not formed. In view of the temperature range under consideration (300-500°F) and the poor mechanical strength of unirradiated 6061-0 aluminum in this range, the importance of the boehmite formation must be assessed completely.

During reactor operation, the unfueled side-plate support structure in the ATR fuel element will operate at a temperature substantially less than the fuel plates. Hence, a compressive load upon the fuel plates may result. If the resultant stresses exceed the currently accepted criterion (the yield stress of the unirradiated cladding material), measurements of those physical properties must be made which will allow evaluation of irradiated fuel plate behavior when subjected to compressive loads.

The necessary thermal stress calculations have been made and show that the fuel plate operating at the maximum power density in reference ATR operation (ie, 250 MW, 40-50-60 MW power unbalance for seventeen days)^[18] may have certain regions in which the 0.2 percent offset yield stress of 6061-0 aluminum is exceeded. These results together with the lack of information upon fuel plate behavior when subjected to compressive loads appear to limit the operable ATR power to 200 MW, with 40 MW in each of the five lobes. Additional means by which reference conditions may be achieved should be investigated; among these are the following:

- (1) Corrosion inhibition by coating the cladding with a corrosion resistant material, such as nickel
- (2) Use of higher strength alloys such as 6061-T6 aluminum
- (3) Use of aluminum alloys with lower corrosion rates such as 2219 (this alloy can also be heat treated to provide better physical properties in comparison to 6061-0)
- (4) Varying the fuel and burnable poison content between plates (radial zone loading) and possibly within plates (azimuthal loading), thus obtaining decreased radial peaking factors (in comparison to the reference fuel).

The in-pile performance of the fuel and cladding materials for each of the four possible solutions must be investigated.

In addition, it is recommended that the PAED-G-12 ETR Water Loop be revised to allow operation with beginning-of-life sample fuel plate temperatures up to 425°F. This can be achieved by increasing the temperature of the coolant entering the sample fuel train. The operating pressure of the loop should be raised to prevent nucleate boiling on the fuel plate surfaces and to provide an adequate margin to thermal burnout. This recommendation is made since it is recognized that there is a finite probability (of the order of 3 percent) that the beginning-of-life surface temperatures in ATR will be near 425°F, and fuel and

cladding material performance at these conditions should be evaluated to assure satisfactory operation of the reactor.

Since the temperature at which the fuel plate forms blisters has been determined by post-irradiation annealing experiments in which the plate is in an isothermal environment, it is recommended that both the reference fuel (U_3O_8) and its more attractive substitute (UAl_3) be irradiated at temperatures up to about 650°F to assure that the effect of temperature during irradiation does not substantially lower the conditions at which blisters form.

Finally, it is recommended that Nb-coated spherical $\text{UO}_2\text{-ZrO}_2$ dispersed in X8001 aluminum not be used in high-performance test reactor fuel plates. This material may be quite satisfactory at lower temperatures and fission densities, but its fabrication costs compared to other fuels make its use unattractive.

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