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ARMY GAS-COOLED REACTOR SYSTEMS PRORAM

HIGH TEMPERATURE IRRADIATION OF UO_2-BeO BODIES

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A SUBSIDIARY OF AEROJET-GENERAL CORPORATION
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ARMY GAS-COOLED REACTOR SYSTEMS PROGRAM

HIGH-TEMPERATURE IRRADIATION OF UO_2-BeO BODIES

by

G. W. Titus, Aerojet-General Nucleonics

J. H. Saling, Battelle Memorial Institute

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ARMY GAS-COOLED REACTOR SYSTEMS PROGRAM

HIGH-TEMPERATURE IRRADIATION OF UO_2-BeO BODIES

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ABSTRACT

HIGH-TEMPERATURE IRRADIATION OF UO_2-BeO BODIES*

G. W. Titus, Aerojet-General Nucleonics

J. H. Saling, Battelle Memorial Institute

As a part of the ML-1 reactor fuel evaluation program, UO_2-BeO bodies containing two UO_2 compositions, 70 and 80 wt% UO_2, were irradiated to a burnup equivalent to 8750 hours of ML-1 reactor operation (8.5% U-235). It was estimated that maximum cladding surface temperatures of 1710°F were attained during irradiation. Reference design burnup for the ML-1 is 9.7% U-235, in 10,000 hours of operation at maximum clad surface temperatures of 1750°F. Post-irradiation examination of the test specimens revealed that the cladding of one of the 80 wt% UO_2-BeO specimens had ruptured after severe swelling. All other specimens showed little external effect from the irradiation. Fission gas release from the fuel varied between 0.59 and 2.7% except for the failed specimen which released about 69%. Considerable change was observed in the microstructure of the irradiated specimens although subsequent X-ray diffraction examination did not indicate serious damage to the crystal structure of either the BeO or UO_2. The data obtained from this experiment are in substantial agreement with that determined in irradiation experiments performed at other facilities (Refs. 1 and 2).** The performance of 70 wt% UO_2-BeO was considered to be suitable for use in the ML-1 reactor.[2] This work was performed under Contract AT(10-1)-880 between Aerojet and the U. S. Atomic Energy Commission.

*Published by Aerojet-General Nucleonics, San Ramon, California

**References are listed at the end of this report.

ARMY GAS-COOLED REACTOR SYSTEMS PROGRAM

HIGH-TEMPERATURE IRRADIATION OF UO_2-BeO BODIES*

INTRODUCTION

GENERAL:

The ML-1 is a prototype gas-cooled, mobile nuclear power reactor system designed by Aerojet under contracts with the AEC Division of Reactor Development and the U. S. Army. Reactor fuels selected for the prototype ML-1 fuel elements were 70 wt% UO_2-BeO and solid UO_2. These fuels are placed in Hastelloy X fuel pins arranged in two concentric rings (Figure 1). The UO_2-BeO fuel is contained in the outer ring of pins; the inner ring of pins contains solid UO_2 fuel. As the hot spot temperature of the fuel elements was calculated to be 1750°F, irradiation of these fuels was performed at about this temperature.

Static capsule irradiations were performed on both fuels to obtain 10,000 ML-1 operating hours equivalent burnup data under close to reactor temperatures. The U-235 burnups anticipated were 3.5 atom% U-235 (UO_2); 9.5 atom% U-235 (70 wt% UO_2-BeO). To obtain this information in a reasonable length of time, the burnup was accelerated to be available in approximately 3000 hours of reactor operation. (The UO_2 irradiation was reported earlier in Ref. 3**)

The irradiation experiment reported here was performed under the joint supervision of personnel from Battelle Memorial Institute (BMI) and Aerojet-General Nucleonics (AGN). The capsule was designed and fabricated by BMI, the specimens by AGN, capsule irradiation was performed in the MTR, and the post irradiation examination in the Battelle hot cell facility.

Results of this experiment indicate no major adverse effects upon the fuel as a result of high temperature, long duration irradiation.

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** References are listed at the end of this report

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THE ML-1 FUEL ELEMENT

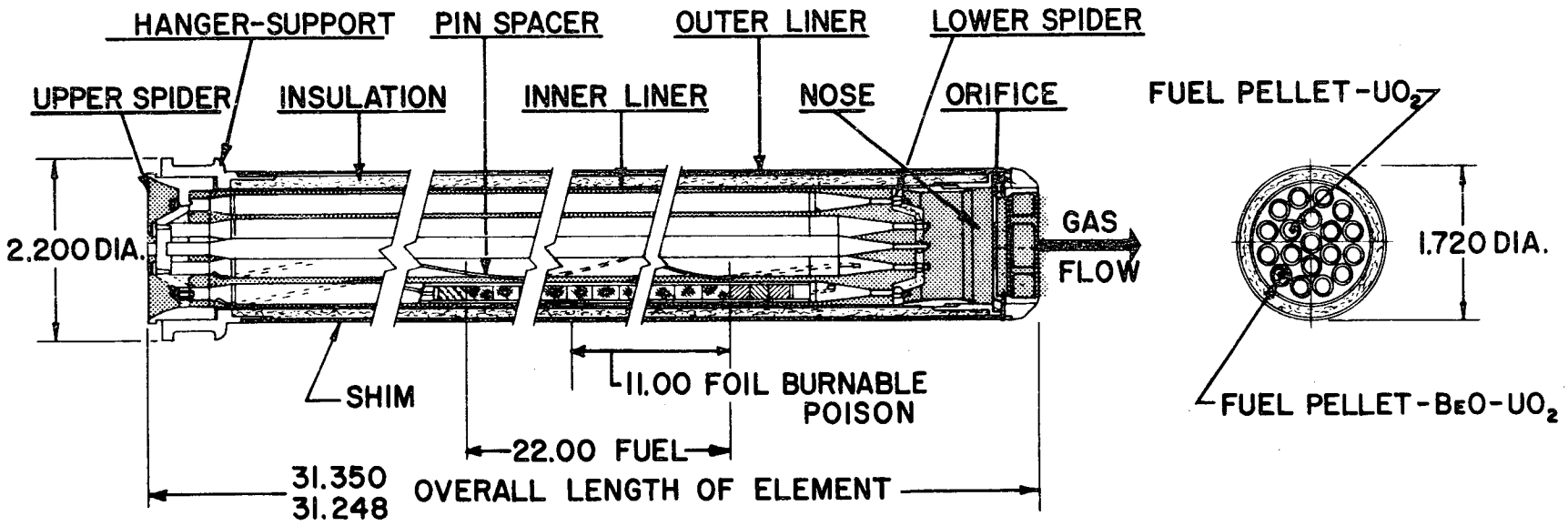


FIGURE 1

EXPERIMENTAL PROCEDURE:

Irradiation specimens were prepared at AGN using fully enriched (Spencer Chemical Co.) UO_2 of 5 to 40 micron particle size and Brush Beryllium grade LOH beryllia powder of 1 to 3 micron particle size. Pre-dried, homogenized ceramic BeO and UO_2 powders were blended two hours in V blenders, wet mixed in a solution of water, castor oil and CD108, then partially dried in a vacuum drying oven. The partially dried material was granulated by being passed through a double screen and finally dried and the fines removed. Small granules of approximately 20 mesh were formed in this treatment. Pellets were cold pressed from these granules in a Stokes F-4 press, then sintered in hydrogen at 3000°F for two hours. After the pellets were cool, they were inspected for integrity and dimensional control. Pellet densities of 96-97% were obtained from this process. Typical chemical analyses of the UO_2 and BeO powders are shown in Tables 1 through 3 below:

TABLE 1

TYPICAL SPECTROGRAPHIC ANALYSIS OF UO_2 CERAMIC GRADE POWDER FROM SPENCER CHEMICAL CO.

Element	ppm	Element	ppm
Cadmium	0.1	Iron	30
Silicon	100	Nickel	10
Boron	1	Aluminum	10
Phosphorous	50	Molybdenum	3
Manganese	2	Tin	1
Magnesium	< 5	Copper	3
Lead	2	Silver	0.1
Chromium	< 5		

TABLE 2

TYPICAL ANALYSIS OF LOH GRADE BeO

SUPPLIED BY BRUSH BERYLLIUM CO.

Element	ppm	Element	ppm
Boron	1.9	Manganese	130
Aluminum	130	Nickel	50
Chromium	15	Sodium	3000
Iron	20	Calcium	50
Lithium	50	Silicon	330
Magnesium	< 500		

TABLE 3

TYPICAL ANALYSIS OF UO_2-BeO CERAMIC

FUEL PELLETS (SPECTROSCOPIC)

Element	ppm	Element	ppm
Silver	ND < 1	Lithium	200
Aluminum	250	Magnesium	10
Boron	ND < 1	Manganese	40
Barium	ND < 10	Nickel	20
Bismuth	ND < 5	Lead	ND < 10
Calcium	ND < 10	Silicon	ND < 10
Cobalt	ND < 1	Tin	150
Chromium	ND < 10	Strontium	ND < 10
Copper	10	Zinc	ND < 20
Iron	5		

ND = Not Detected

I. PREPARATION OF THE EXPERIMENT

A. FUEL SPECIMEN PREPARATION

The irradiation capsule employed a standard double-walled design utilizing a helium heat transfer barrier in the annulus between the walls. Six irradiation specimens supported by a nickel expanded metal basket were aligned vertically in NaK and sealed into the inner capsule. The irradiation specimens were prepared at AGN utilizing fully enriched 70 and 80 wt% UO_2-BeO pellets. Each specimen assembly contained 1.056 inches of fuel, consisting of five 70 or 80 wt% UO_2-BeO pellets, 0.145 inches in diameter, 0.211 inches long enclosed in a 0.240 inch outside diameter, 0.030 inch wall Hastelloy X tube specimen (Figure 2). Specimen ends were closed under a helium atmosphere with press fit Hastelloy X plug machined from wrought Hastelloy X rod. Each plug was heliarc welded in place and the final assembly leak checked after welding. A two to three mil radial gap was present between the outside of the pellets and the inside of the cladding because of dimensional variations. Thin sectioned, stainless steel spacers prevented the pellets from touching the end plugs. A radiograph of several specimens assembled into the capsule, prior to irradiation, is shown in Figure 3.

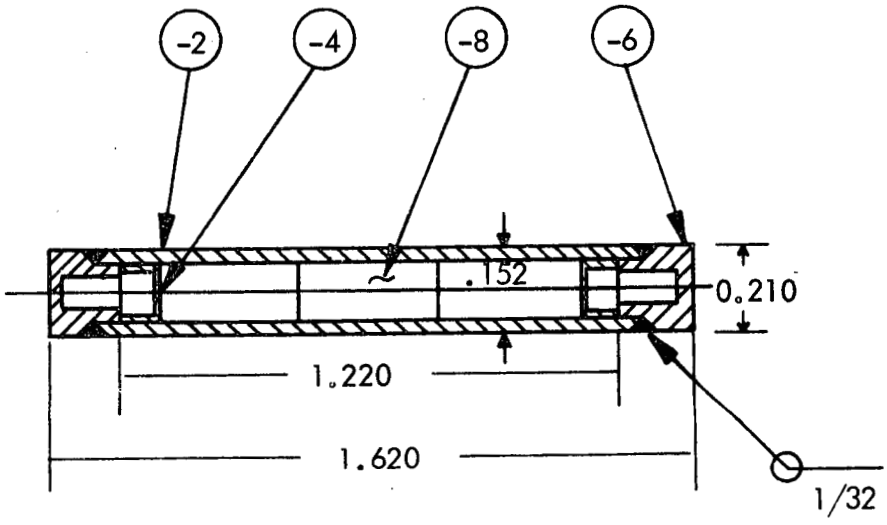
The specimens assembled into this experiment were of two fuel compositions, four of 70 wt% UO_2-BeO and two of 80 wt% UO_2-BeO; the 70 wt% UO_2 specimens contained 1.24 grams of U-235; the 80 wt% UO_2 specimens contained 1.74 grams.

B. IRRADIATION CAPSULE DESIGN

The irradiation capsule was designed to maintain the cladding-surface temperature of the peak-flux specimen at 1725°F by a combination of fission, gamma, and electrical heating. Standard double-wall construction of AISI Type 347 stainless steel was employed. The specimens were suspended in a nickel basket immersed in NaK. Details of the capsule are shown in Figure 1 and an assembled view is shown in Figure 4.

The capsule inner shell was 0.438 inch in inside diameter, 0.900 inch in outside diameter, and 16-3/4 inches in inside length. Three 1/16 inch outside diameter stainless steel-sheathed magnesium oxide-insulated Kanthal heater elements were brazed into helical grooves machined on the outer surface

CAPSULE - UO_2 SPECIMEN



NO. REQ'D	DET. NO.	
3	8	PELLET - UO_2
2	6	CAP - 1/4 DIA. HASTELLOY "X" ROD
2	4	SPACER - 1/8 DIA. CRES TYPE 316 MIL - S - 7720, COND. A
1	2	SHELL - .210 O.D. X .031 WALL HASTELLOY "X" TUBING

FIGURE 2 11.5-63-287

RADIOGRAPH OF THE INNER SHELL OF CAPSULE
MTR GCR-3 WITH SPECIMENS IN PLACE

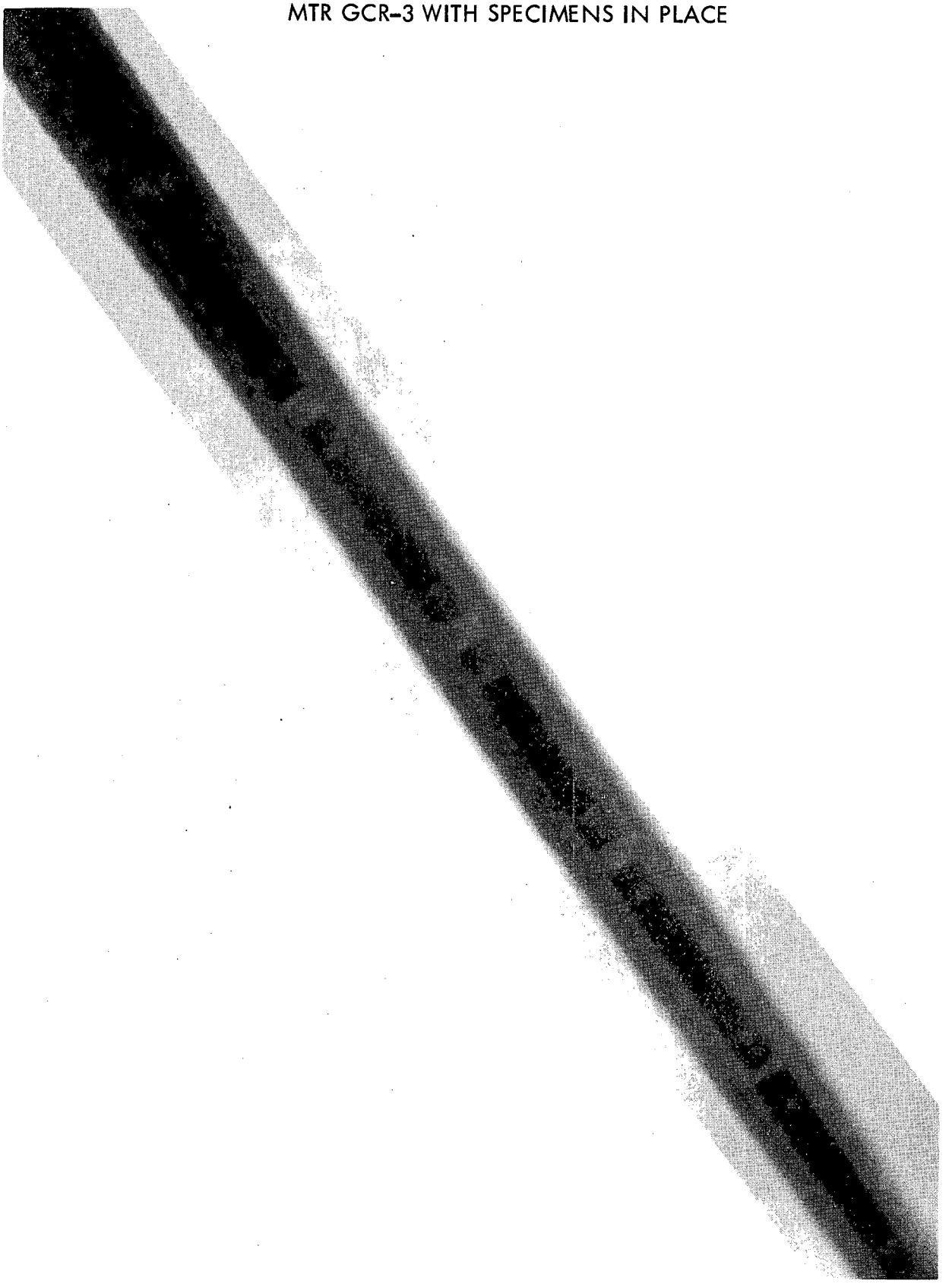


FIGURE 3 11.5-63-053

DETAILS OF CAPSULE MTR GCR-3

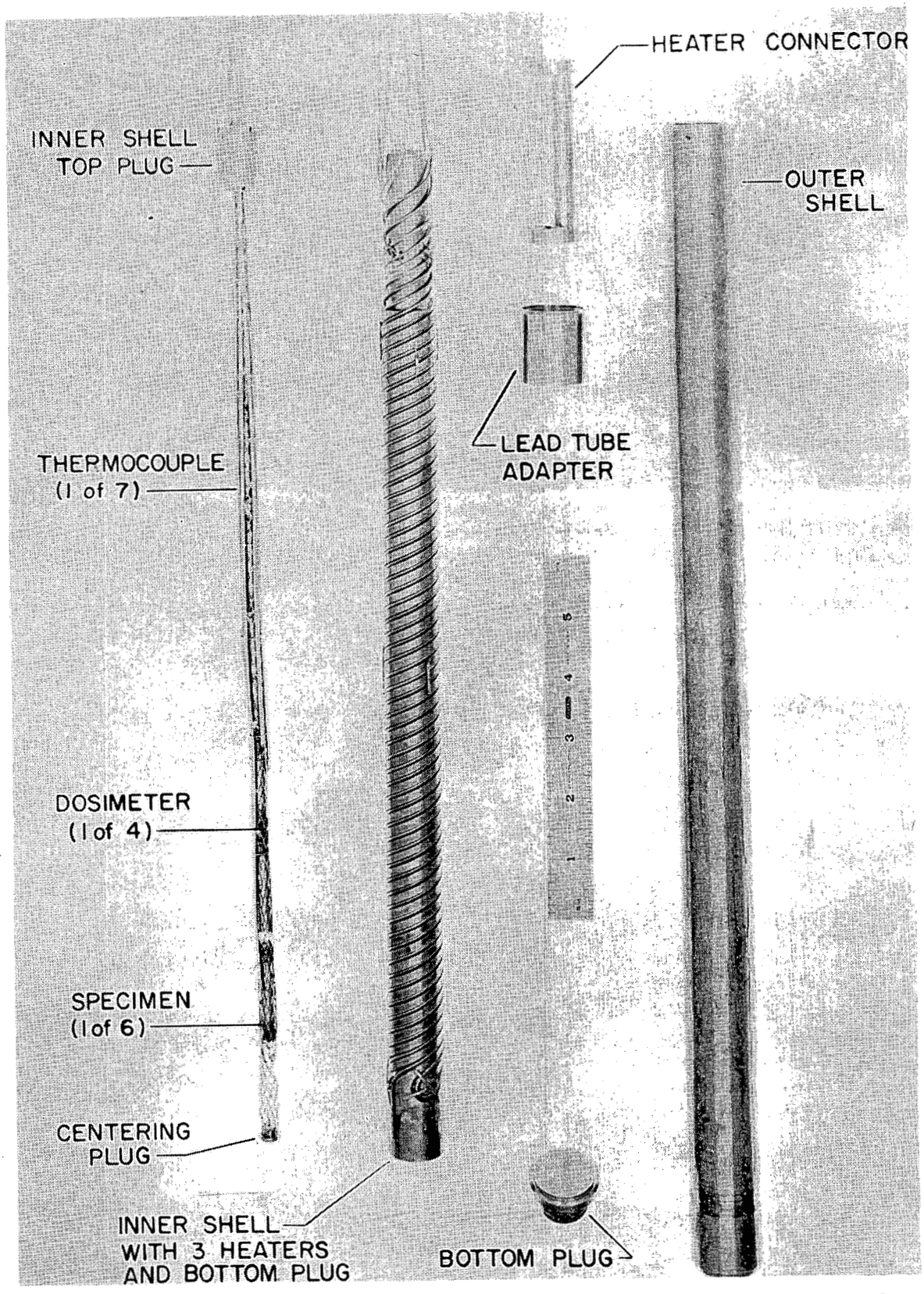


FIGURE 4 11.5-63-054

of the inner shell. Each heater was rated at 1 kw. A bottom plug, machined to a 1/8-inch wall thickness to facilitate gas sampling after irradiation, was welded to the inner shell. A recess was machined into this plug to engage a fitting on the bottom of the specimen basket to maintain axial alignment of the basket. The header was welded to the inner shell in a helium-filled dry box after the specimens and NaK were loaded. The thermocouple passed through a brazed double seal in the heater. At this point the inner shell was radiographed (Figure 2) and leak checked.

The AISI Type 347 stainless steel capsule outer shell was 0.970 inch in inside diameter, 1.125 inches in outside diameter, and 18 inches in inside length. The header of the inner shell was torch brazed to the inside surface of the outer shell. Heater leads passed through slots around the periphery of the header. The brazed joint sealed the helium annulus from the lead tube. The 1-inch outside diameter AISI Type 304 stainless steel lead tube was joined to the capsule by an adapter welded to the top of the capsule. The final closure of the outer capsule was made by welding the bottom to the outer shell in a helium-filled dry box. This weld was also checked with a helium-leak detector.

Six of the seven thermocouples in the capsule were positioned individually at the midpoint of each specimen. The seventh was located at the upper end of the fueled section of the fourth specimen from the top (Specimen 6) in an attempt to determine temperature differences in the specimen resulting from flux perturbation. The thermocouple on the third specimen from the top (Specimen 4) was inoperative after the capsule was assembled. In all cases the thermocouple beads were separated from the cladding surfaces by about 1/32 inch and were estimated to indicate temperatures about 35 to 40 F^{o} below the cladding temperatures. The thermocouples were passed up into the lead tube through the heater junction and were joined to standard thermocouple leadout wire with special connectors.

The leads from each of the three 1-kw capsule heaters were connected to individual 16-gage copper leadouts in an Al_2O_3 packed junction in the lead tube. The copper leadouts were contained in a 1/4-inch-outside diameter AISI Type 304 stainless steel sheath packed with magnesium oxide. The capsule heaters were designed to meet all requirements for electrical heating with two of the three heaters operative.

II. IRRADIATION HISTORY

The irradiation of this capsule (designated as MTR-GCR-3) began in MTR core position A-27-NE at the beginning of cycle 145. The requested neutron flux for this position was 8.5 x 10^{13} nv, the quoted flux was 4-8 x 10^{13} nv. Startup for this cycle was 19 September 1960. The capsule was initially placed at the bottom of the position to provide flexibility for movement to a higher flux by raising the capsule in the position. As the actual specimen temperatures were below the desired operating levels (see Table 4) the capsule was raised 7 inches during the subsequent reactor shutdown. The thermocouple on specimen 4 (the third specimen from the top) was inoperative throughout this cycle (Table 4).

It became apparent early in cycle 146 that movement of the capsule did not greatly improve the specimen temperature pattern so that the auxiliary heat required to maintain the temperature was considerably more than anticipated. Transfer of the capsule to a higher flux position was requested and scheduled to correct this situation. Shortly after deciding to transfer, however, all specimen thermocouples but one failed, and it was decided not to transfer the capsule as movement might cause the remaining thermocouple to fail.

Shortly after the beginning of cycle 147 the remaining thermocouple failed and the auxiliary heat input had to be greatly reduced.

During the shutdown following cycle 147, efforts were made to re-establish operation of the thermocouples. This effort was partially successful as two thermocouples operated during cycle 148. One heater failed during cycle 148 so that the remaining heaters were operated at reduced power to maintain heater life. Peak-flux specimens attained a surface temperature of approximately 1700°F in cycle 149 with aid of 2000 watts from the auxiliary heaters. Attempts after this cycle to restore the inoperative thermocouples were unsuccessful.

Another heater failed at the beginning of cycle 150 and the remaining heater had insufficient output to maintain desired temperature levels. As the heater-control thermocouple also failed, the remaining heater was not operated for the duration of this cycle. A surface temperature of 1550°F without auxiliary heatwas estimated for the peak-flux specimen based upon extrapolation of previous temperature data.

TABLE 4

SUMMARY OF TEMPERATURE DATA FOR MTR GCR-3 IRRADIATION EXPERIMENT

MTR Cycle	Full Power Days	Average Electric Heat Input, Watts	Specimen Clad Surface Temperature, °F													
			3			11			6			13			10		
			Mean	Max.	Min.	Mean	Max.	Min.	Mean	Max.	Min.	Mean	Max.	Min.	Mean	Max.	Min.
145	18.2	1550	1425	1535	1375	1550	1685	1480	1420	1550	1330	1430	1580	1330	1275	1385	1160
146	10.0	2200	1510	1570	1445	1670(1)	1685	1655	1570(2)	1600	1525	1440(3)	1465	1415	1485(3)	1540	1425
147	18.0	2300	1450(4)	--	--	1610(6)	--	--	1520(6)	--	--	1400(6)	--	--	1440(6)	--	--
148	17.3	740	1310	1375	1280	1480(6)	--	--	1380(6)	--	--	1280(6)	--	--	1300(6)	--	--
149	25.6	2000	1540(6)	--	--	1710(6)	--	--	1600(6)	--	--	1475	1515	1445	1510(6)	--	--
150(5)	10.5	--	1380(6)	--	--	1550(6)	--	--	1450(6)	--	--	1325	1335	1310	1350(6)	--	--
151	14.5	--	1380(6)	--	--	1550(6)	--	--	1450(6)	--	--	1325(6)	--	--	1350(6)	--	--
152	16.8	--	1380(6)	--	--	1550(6)	--	--	1450(6)	--	--	1325(6)	--	--	1350(6)	--	--
153	16.5	--	1380(6)	--	--	1550(6)	--	--	1450(6)	--	--	1325(6)	--	--	1350(6)	--	--
154	15.5	--	1380(6)	--	--	1550(6)	--	--	1450(6)	--	--	1325(6)	--	--	1350(6)	--	--

(1) This thermocouple failed three days after the start of the cycle.

(2) This thermocouple failed six days after the start of the cycle.

(3) These thermocouples failed seven days after the start of the cycle.

(4) This thermocouple failed shortly after the reactor startup. The heaters were turned off for the remainder of the cycle.

(5) Heaters failed shortly after reactor startup and remaining thermocouple failed during cycle.

(6) Estimated on the basis of previous thermocouple data.

During the remaining four irradiation cycles, no information on specimen temperature was available and the remaining heater was not operated. The estimates of specimen temperatures (shown in Table 4) were based upon earlier thermocouple data as there was no reason to suspect gross changes in flux.

The capsule was discharged at the termination of cycle 154, permitted to decay two weeks, and shipped to the BMI Hot Cell Facility at Columbus, Ohio.

Capsule operation left much to be desired as design specimen temperatures were attained for only a small portion of the total irradiation time. However, the estimated cladding temperatures were maintained close to ML-1 average cladding temperatures ($\sim$1550°F), even though hot spot conditions were not duplicated. Fuel central temperatures were considerably higher than those predicted in the ML-1 fuel element because of burnup acceleration.

III. POST IRRADIATION EVALUATION

Post irradiation evaluation of this capsule began shortly after the capsule arrived at BMI. The examination included the following:

1) Visual and dimensional examination,
2) Gamma scanning,
3) Measurement of fission product gas release,
4) Burnup analyses,
5) Metallographic examination,
6) Chemical analyses, and,
7) X-ray diffraction analyses.

A. CAPSULE OPENING AND GAS SAMPLING

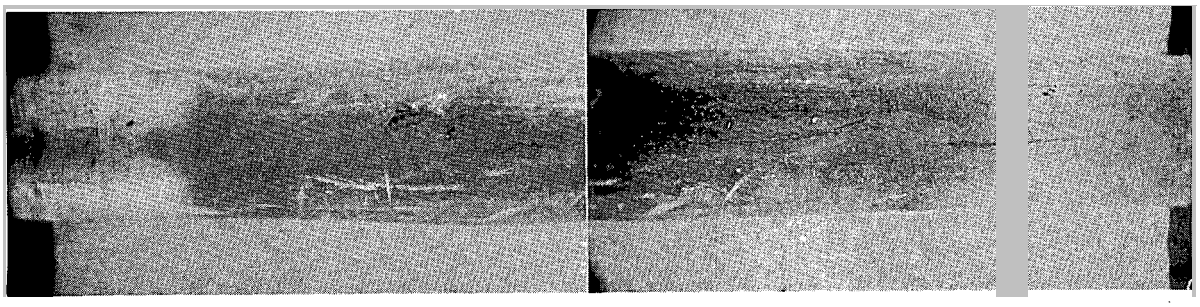
The outer shell of the capsule was removed using a remotely operated pipe cutter. The inner capsule shell was then punctured and sampled for fission product gases released by specimens during irradiation and the gases collected. There was evidence of fission product gas release, indicating that one or more specimens had failed during irradiation.

The top of the inner capsule shell was next removed and the inner shell immersed in butyl alcohol. This step was performed to react the NaK heat transfer fluid and remove it from the inner shell. Following this step, the nickel specimen support baskets, with specimens still attached, were removed and disassembled. Each specimen was cleaned and identified in preparation for visual examination.

B. VISUAL EXAMINATION

Each specimen was rinsed in fresh butyl alcohol, dried and thoroughly examined through a stereo microscope. In general, all the specimens appeared intact and unchanged except specimen 11 (Figure 5). This specimen contained 80 wt% UO_2-BeO and the cladding showed a longitudinal rupture, 1/4 inch long over the fuel section. An unfailed specimen considered typical of the other irradiation specimens is shown in Figure 6. No swelling or cracking could be detected in the claddings of the other five specimens.

SPECIMEN 11 CONTAINING BeO-80 WT % UO_2 IRRADIATED TO AN AVERAGE BURNUP OF 5×10^{20} FISSIONS/cc AT A MAXIMUM CLAD SURFACE TEMPERATURE OF 1750°F IN CAPSULE MTR GCR-3



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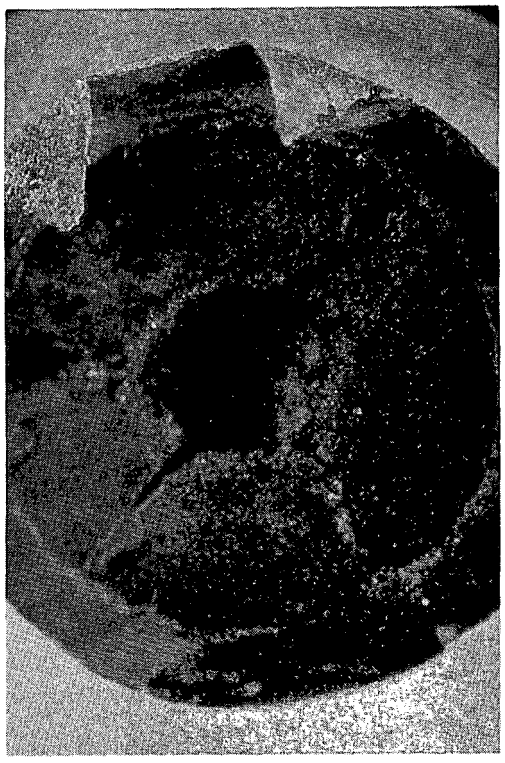
(a) Overall view of Specimen 11 showing swelling and rupture. Note the discolored area over the fuel section



12X

12X

(b) View of transverse and longitudinal cross sections of Specimen 11 showing central void in the fuel

FIGURE 5

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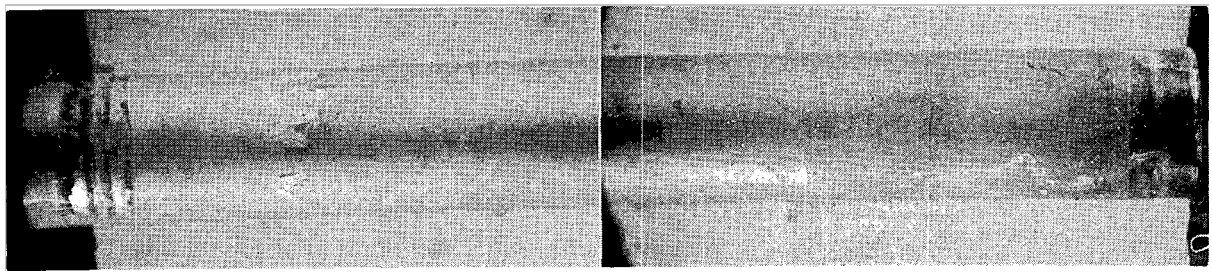
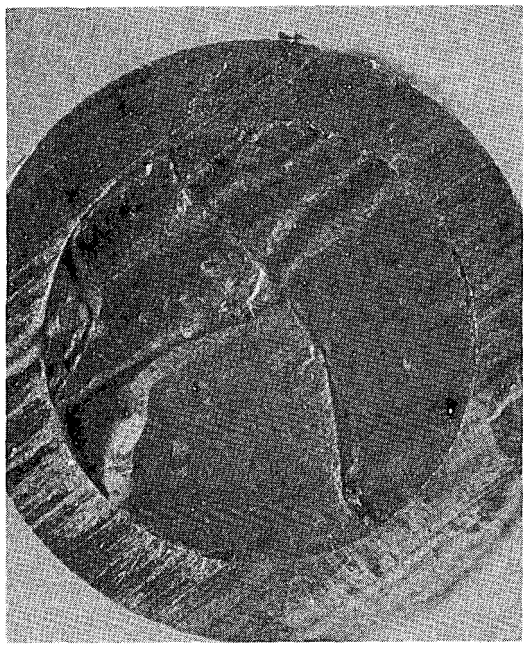
TYPICAL CONDITION OF UNFAILED SPECIMENS CONTAINING BeO-70 wt % UO_2 FROM CAPSULE MTR GCR-3



3X

(a) Specimen 10 irradiated to a burnup of 5.8×10^{20} fissions/cc at a maximum clad surface temperature of 1510°F



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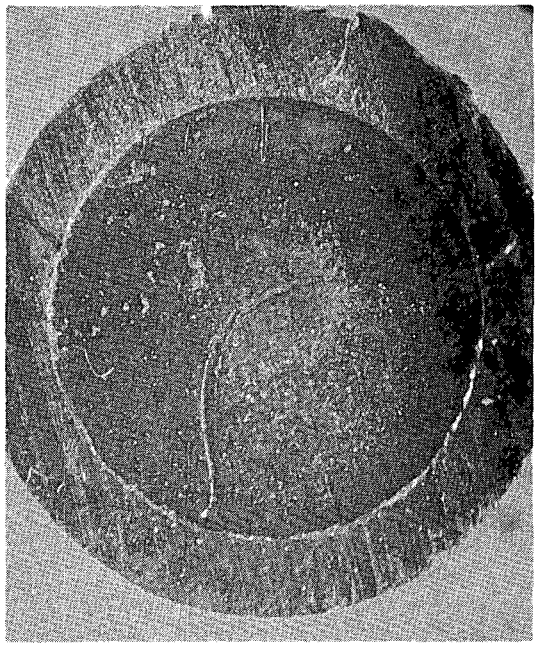
(b) Specimen 10, transverse section, irradiated to a burnup of 5.8 X 10^{20} fission/cc at a maximum clad surface temperature of 1510°F.



12X

(c) Specimen 6, transverse section, irradiated to a burnup of 6.4 X 10^{20} fissions/cc at a maximum clad surface of 1600°F.

FIGURE 6

11.5-63-056

C. GAMMA SCANNING

As an initial step in determining specimen burnup, the relative intensity of gamma radiation emitted from each pin was determined as a function of length. The data from this operation was correlated with the analytically determined burnup (discussed later) to yield the relative burnup distribution over the length of the individual fuel specimen as well as the vertical neutron flux distribution along the capsule length. The 0.717 Mev zirconium 95 photo peak was used to obtain the gamma intensity profile.

D. MEASUREMENT OF PHYSICAL DIMENSIONS

The length and diameter of each specimen were measured before and after irradiation. Dimensions were measured with standard friction thimble micrometers. The accuracy of these measurements has been stated as $\pm$ 0.0005 inches. The data obtained from the present specimens are displayed in Table 5. From the data it is evident that very little change could be detected between pre- or post-irradiation measurements except for specimen 11. Considerable swelling was apparent in visual examination of this specimen. This swelling averages 15% diametral increase, 16% maximum.

E. GAS SAMPLING OF IRRADIATION SPECIMENS

After length and diameter measurements were completed, the fission product gases were removed from each specimen except specimen 11, and collected for analysis.

Gamma-ray and mass spectrographic techniques were used in analyzing the specimens. The percent of gas released from the fuel specimen by fission fragment recoil was calculated.

One milliliter samples were collected in glass vials for gamma ray spectrography; four samples were analyzed for each specimen. In the analysis technique employed, the intensity of the characteristic 0.51 Mev gamma ray emitted during the radioactive decay of krypton-85 was measured. A gamma-ray spectrometer having a well-type NaI(Th) crystal was used in making the measurements.

For mass spectrographic analysis, 100-milliliter samples were collected in glass vials. In this analysis only one sample was analyzed for each specimen. Isotopic ratios for krypton-83, krypton-84, krypton-85, and krypton-86 as well as xenon-131, xenon-132, xenon-134 and xenon-136 were established. From these data, the quantity of each isotope present was established and the fraction of fission gas released from each specimen calculated.

The results of these analyses are shown in Table 6. The gas release in general was small except for specimen 11 where cladding rupture and possibly central core melting had occurred. All of the fission gas collected from the inner shell of the capsule was assumed to have originated from specimen 11.

TABLE 5

CHANGES IN PHYSICAL DIMENSIONS AND WEIGHTS OF HASTELLOY X-CLAD 70 wt% UO_2-BeO AND 80 wt% UO_2-BeO SPECIMENS IRRADIATED IN CAPSULE MTR GCR-3

Specimen	UO_2 Content, wt%	Preirradiation Physical Properties[a]			Postirradiation Physical Properties[a]			Change in Physical Properties[a]		
		Diameter, in.	Length, in.	Weight, g	Diameter, in.	Length, in.	Weight, g	Diameter, per cent	Length, per cent	Weight, per cent
3	70	0.2400	1.6180	7.8570	0.2412	1.6212	7.8606	0.5	0.2	< 0.1
11	80	0.2400	1.5924	8.2268	0.2753	1.5921	8.2940	15[b]	< 0.1	0.8
4	70	0.2400	1.6210	7.9001	0.2412	1.6212	7.9043	0.5	< 0.1	< 0.1
6	70	0.2400	1.6175	7.8555	0.2399	1.6160	7.8550	< 0.1	< 0.1	< 0.1
13	80	0.2405	1.5990	8.1355	0.2407	1.5978	8.1271	< 0.1	< 0.1	0.1
10	70	0.2420	1.6212	7.9152	0.2413	1.6108	7.9164	- 0.5	< 0.1	< 0.1

(a) Average of three readings taken for each measurement.

(b) Maximum swelling which occurred was 16%.

TABLE 6

FISSION GAS RELEASE DATA FROM HASTELLOY X-CLAD 70 wt% UO_2-BeO AND 80 wt% UO_2-BeO SPECIMENS IRRADIATED IN CAPSULE MTR GCR-3

Specimen	UO_2 Content, wt%	Amount of Krypton-85 Released,[a] per cent: Determined by Gamma-Ray Spectroscopy	Amount of Krypton-85 Released,[a] per cent: Determined by Mass Spectroscopy	Estimated Amount of Krypton-85 Released by Recoil, per cent
Capsule	--	69	18	--
3	70	2.8	2.2	0.24
11	80	(Specimen ruptured)	--	0.24
4	70	1.3	1.1	0.24
6	70	1.5	1.2	0.24
13	80	2.8	2.7	0.24
10	70	0.62	0.59	0.24

(a) Calculations were based on a krypton-85 fission yield of 0.293% (Ref. 4) and a branching ratio of 0.36% (Ref. 5)

F. FUEL BURNUP ANALYSIS

The burnup analyses determined were very close to the level established as the 10,000 hour ML-1 burnup value, i.e., 9.7 atom% U-235. These values were determined by performing cesium-137 and isotopic burnup analyses upon irradiated fuel from specimens 4 and 10. Data from these analyses were correlated with the gamma scan performed earlier to obtain a burnup value for each specimen. The results obtained from the burnup analyses and gamma scan correlation are displayed in Table 7.

Approximately half of a fuel specimen was utilized in performing the analyses, the data obtained from such an analysis is an average fuel burnup. However, it should be noted in Table 7 that the ratio of surface to centerline fuel burnup is about three due to the fuel enrichment of these specimens.

The isotopic and cesium-137 analyses were performed by Phillips Petroleum Company, Chemical Processing Plant at NRTS, Idaho.

There was good agreement between the burnup values obtained by radiochemistry (Cs-137) and isotopic analysis. The latter technique is generally considered the more accurate of the two in this instance, as considerable cesium migration can be expected at the fuel temperatures encountered in this irradiation. In this instance, however, the close agreement of these two techniques indicated that little cesium migration had occurred.

Calculated values for the specimen cladding and central fuel temperatures are based upon the fission heat generation rates. In general, these values are slightly higher than temperatures measured during the first two irradiation cycles. This may indicate attainment of higher temperatures than were recorded by the thermocouples.

G. METALLOGRAPHIC SPECIMEN EXAMINATION

This phase of the capsule post-irradiation examination was performed upon transverse and longitudinal sections of specimens 4, 6, 10, (70 wt% UO_2-BeO), 11 and 13 (80 wt% UO_2-BeO). For comparison, transverse sections of selected unirradiated specimens mounted in "Col-Weld" were examined.

The unirradiated specimens were prepared for microscopic examination by grinding on 240, 400 and 600 grit silicon carbide paper, followed by polishing on a high speed polishing wheel covered with lintless cloth. Polishing was performed in a slurry of Linde A, 3% Cr_2O_3 and water. These specimens were examined in the as-polished condition and after swab etching with a solution composed of 25 grams NH_4FHF and 75 grams of hot water.

The irradiated sections were mounted in Bakelite with resinoid added to aid in fuel retention. After mounting, each specimen was ground on two 240 grit papers with the specimen under 40 pounds pressure, followed by grinding on two 400 grit papers under 30 pounds pressure. Final grinding was accomplished on a single 600 grit paper with 20 pounds applied to the specimen.

All grinding was accomplished with water lubricant. Initial polishing was performed on one micron diamond with kerosene lubricant; final polishing

TABLE 7

IRRADIATION CONDITIONS AND EFFECTS OF HASTELLOY X CLAD

70 AND 80 wt% UO_2-BeO FUEL PINS IN CAPSULE MTR GCR-3

Specimen Number	UO_2 Content, wt%	Specimen Clad Surface Estimated Range of Temperature, Based on Thermocouple Measurements, °F			Average Specimen Temperature, °F Calculated From Fission Heat Generation Rate[a]		Fission Heat Generation Rate, Btu/hr/ Specimen	Estimated Average Uranium-235 Fission Burnup			
										Average Fissions	
		Min.	Max.	Mean	Clad Surface	Central Core[b]		Cesium-137 Analysis atom%	Isotopic Analysis atom%	Per cc of Fuel Pellet[d] x 10^{20}	Per cc of UO_2[d] x 10^{20}
3	70	1280	1570	1355	1480	2550	1490	8.0[c]	6.9[c]	5.2	13
11	80	1480	1710	1520	1450	2490	1450	5.5[c]	4.8[c]	5.0	9.2
4	70	--	--	--	1720	3020	1810	9.8	8.4	6.3	16
6	70	1330	1600	1425	1740	3060	1835	9.9[c]	8.5[c]	6.4	16
13	80	1310	1580	1380	1640	2860	1700	6.5[c]	5.6[c]	5.9	11
10	70	1275	1540	1325	1610	2810	1670	9.0	7.8	5.8	15

(a) Based on fission and gamma, heat generation rate, adjusted to account for the electrical heat.

(b) Based on a thermal conductivity value of 4 for both 70 and 80 wt% UO_2-BeO.

(c) Obtained by correlating the isotopically determined burnups with gamma-scan profile.

(d) Based upon the isotopically determined burnup. The surface to centerline burnup ratio was calculated to be about 3 with the maximum centerline burnup about 30 x 10^{20} fissions/cc of UO_2.

was done on Microcloth, lubricated with a slurry of 2% Bichromic acid. Each polishing required approximately four hours to complete. A solution of ammonium bifluoride and hydrogen fluoride was used to etch the BeO; a sulphuric acid-hydrogen peroxide mixture was used upon the UO_2; glyceregia was used on the Hastelloy X cladding material.

Of the specimens sectioned, specimens 4 and 6 experienced the highest irradiation temperatures, and specimen 10 operated at the lowest measured temperature. Specimen 11 obviously experienced the highest central fuel temperatures as central melting occurred in this specimen.

Examination of the UO_2-BeO fuel revealed radial and circumferential cracking in all fuel sections observed. The composite views of transverse sections from specimens 6 and 10 shown in Figures 7 and 8 are typical of the cracking found.

Of the 80 wt% UO_2-BeO specimens (11 and 13), specimen 11 was damaged through central melting which resulted in major swelling. This caused failure of the cladding as illustrated in Figure 5. Macrophotographs illustrating the damaged areas are shown in Figure 9 and a longitudinal composite of the melted area is shown in Figure 10. Because of this failure, the other 80 wt% UO_2-BeO specimen was sectioned to investigate specimen integrity. A microphotograph and longitudinal composite section (Figures 11 and 12) clearly show that this specimen appears unaffected by the exposure.

In the microscopic examination of these specimens, initial studies dealt with the unirradiated fuel bodies. Microstructures observed in the 70 and 80 wt% UO_2-BeO bodies (Figure 13) show that relatively uniform dispersions were achieved in both fuel loadings, although the UO_2 phase appears to be the primary phase in the 80 wt% specimen.

In general, the major effects noted were:

1) The agglomeration of the UO_2 into larger and apparently discontinuous particles in the 70 wt% UO_2 bodies,

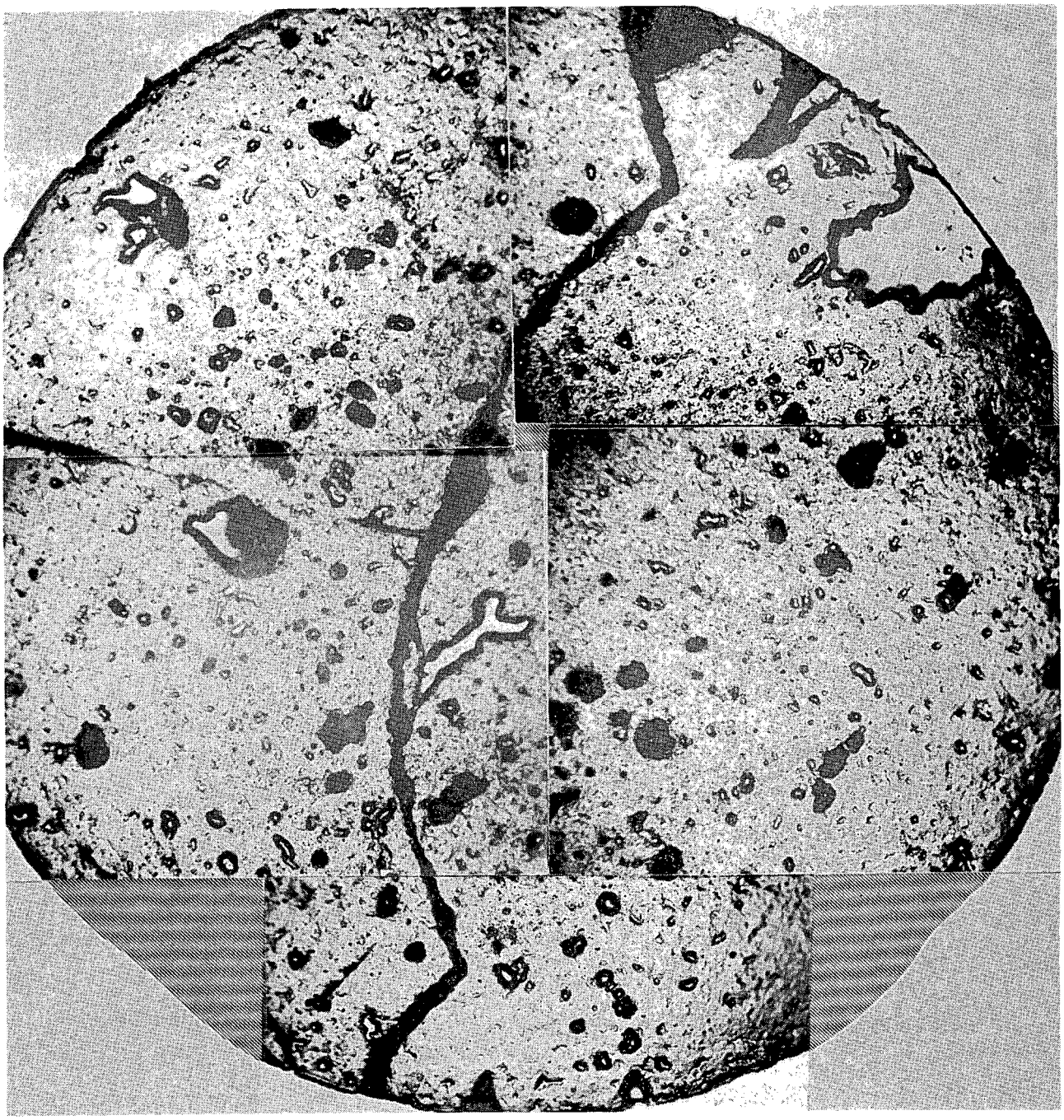
2) Pore formation in UO_2 particles positioned close to the pellet edge and void formation at the particle periphery in particles more centrally located,

3) The BeO microstructure was not greatly changed, although its etching and polishing behavior appeared altered from unirradiated material.

It was difficult to detect a structure in the irradiated BeO through etching. The same general comments apply for 80 wt% UO_2 bodies although the UO_2 particles showed a tendency to stratify and form a continuous phase.

The following significant points concerning the post irradiation condition of the fuel were revealed by a detailed study of specimens containing both fuel loadings: Fuel particles close to the surface of the 70 wt% UO_2 pellets showed a considerable increase in porosity (Figure 13 is the typical pre-irradiation condition, Figure 14 shows the fuel particles after irradiation to 8-9 atom% U-235 burnups). This increase in porosity is considered to have

COMPOSITE OF AN AS-POLISHED TRANSVERSE SECTION OF SPECIMEN 6 (BeO-70 WT % UO_2) IRRADIATED TO A BURNUP OF 6.4×10^{20} FISSIONS/cc AT A MAXIMUM CLAD SURFACE TEMPERATURE OF 1600°F

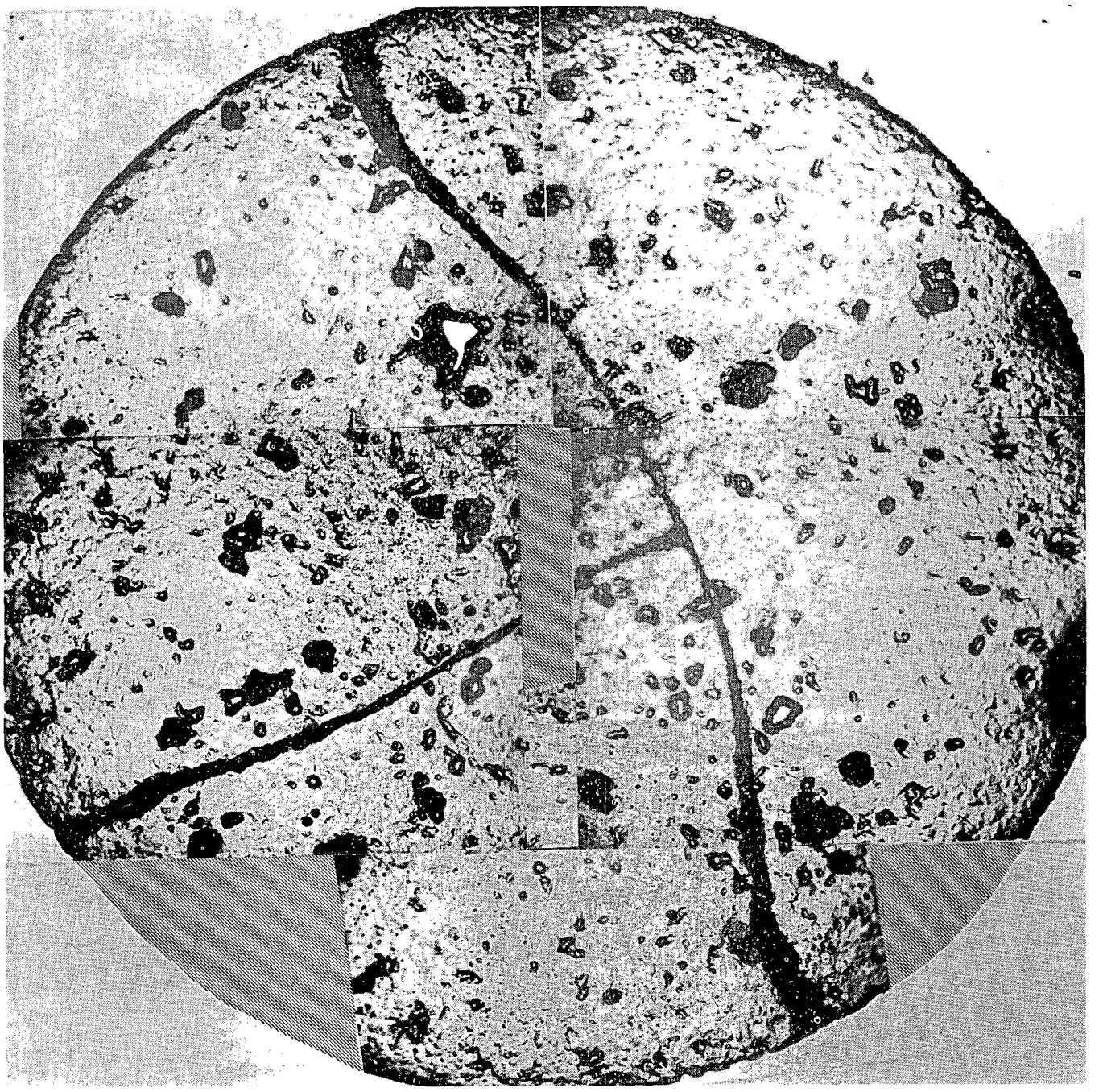


50X

FIGURE 7

115-63-057

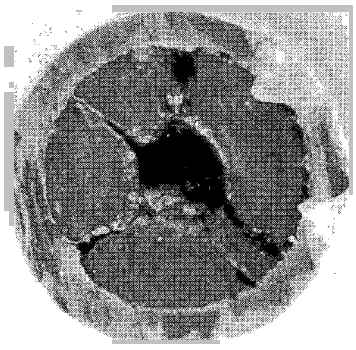
COMPOSITE OF AN AS-POLISHED TRANSVERSE SECTION OF SPECIMEN 10 (BeO-70 WT % UO_2) IRRADIATED TO A BURNUP OF 5.8×10^{20} FISSIONS/cc AT A MAXIMUM CLAD SURFACE TEMPERATURE OF 1510°F



50X
FIGURE 8

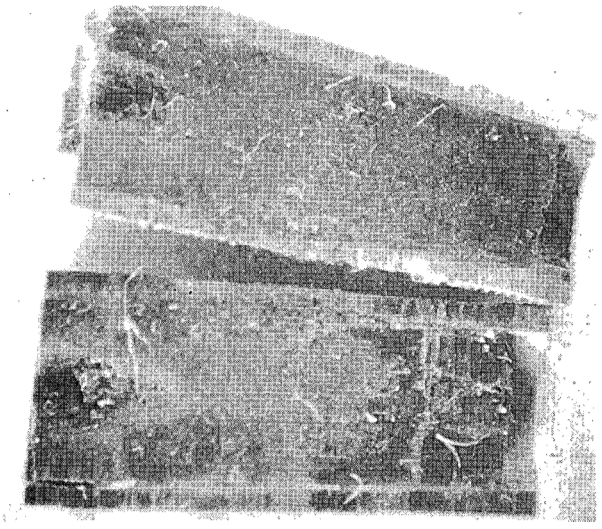
11.5-63-058

CENTRAL VOID IN SPECIMEN 11



6X

a. Transverse section of central void in specimen 11, 80 wt% UO_2-BeO fuel



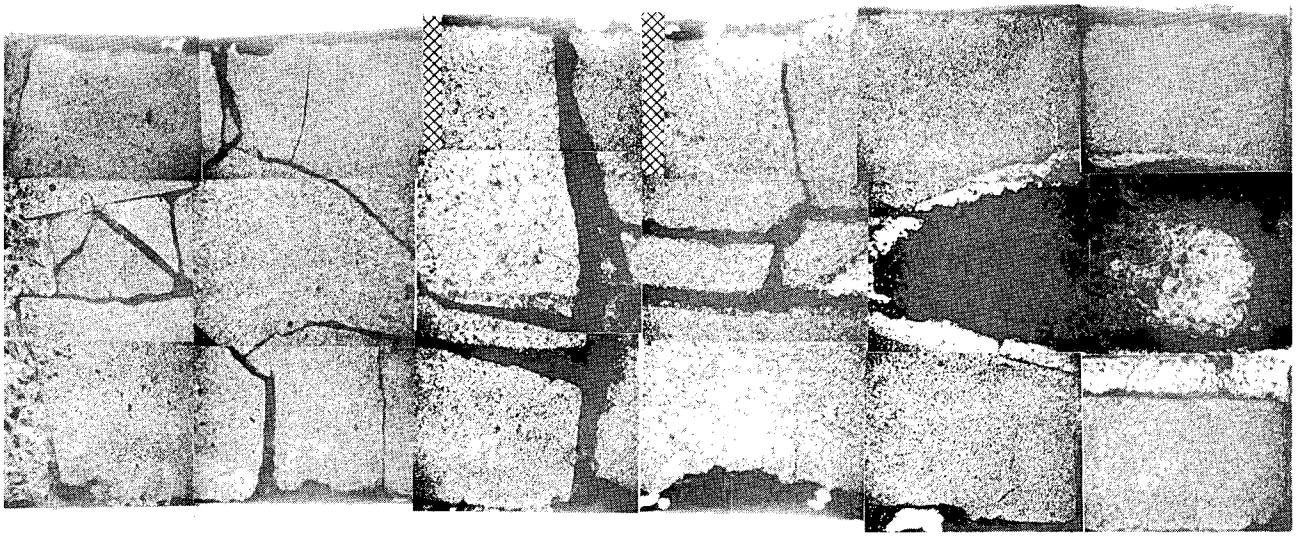
4X

b. Longitudinal view of specimen 11, 80 wt% UO_2-BeO fuel

FIGURE 9

11.5-63-176

LONGITUDINAL SECTION THROUGH SPECIMEN 11



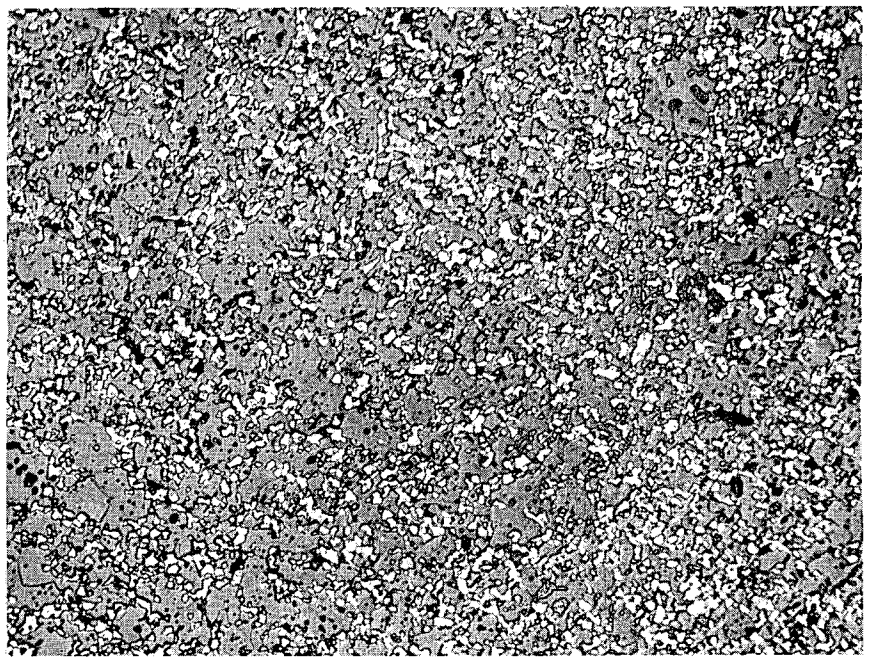
13X

26X

80wt% UO_2 - BeO PELLET, AS POLISHED

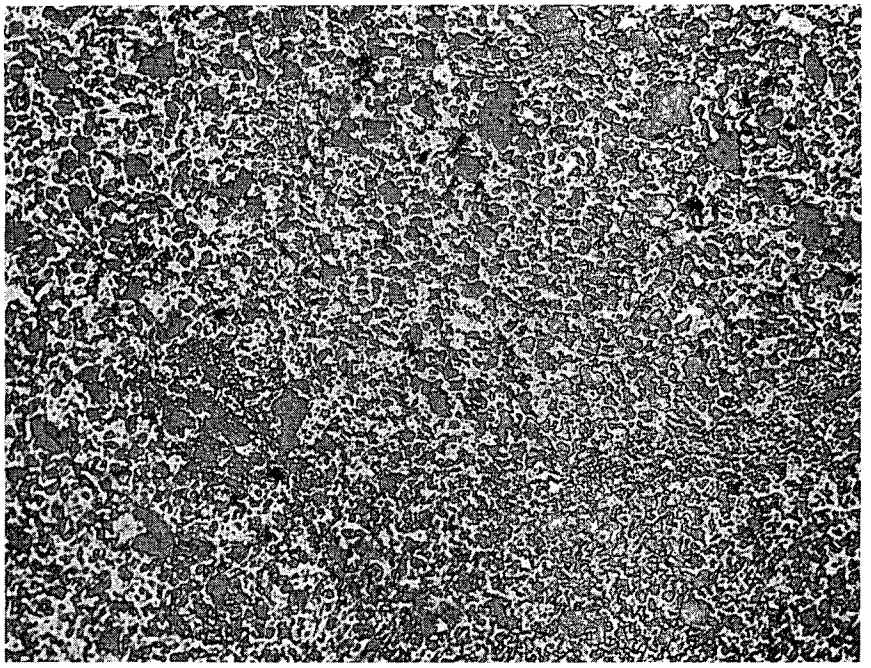
FIGURE 10

PHOTOMICROGRAPHS OF 70 AND 80 wt % UO_2-BeO



250X

70 wt% UO_2-BeO, Unirradiated and etched



250X

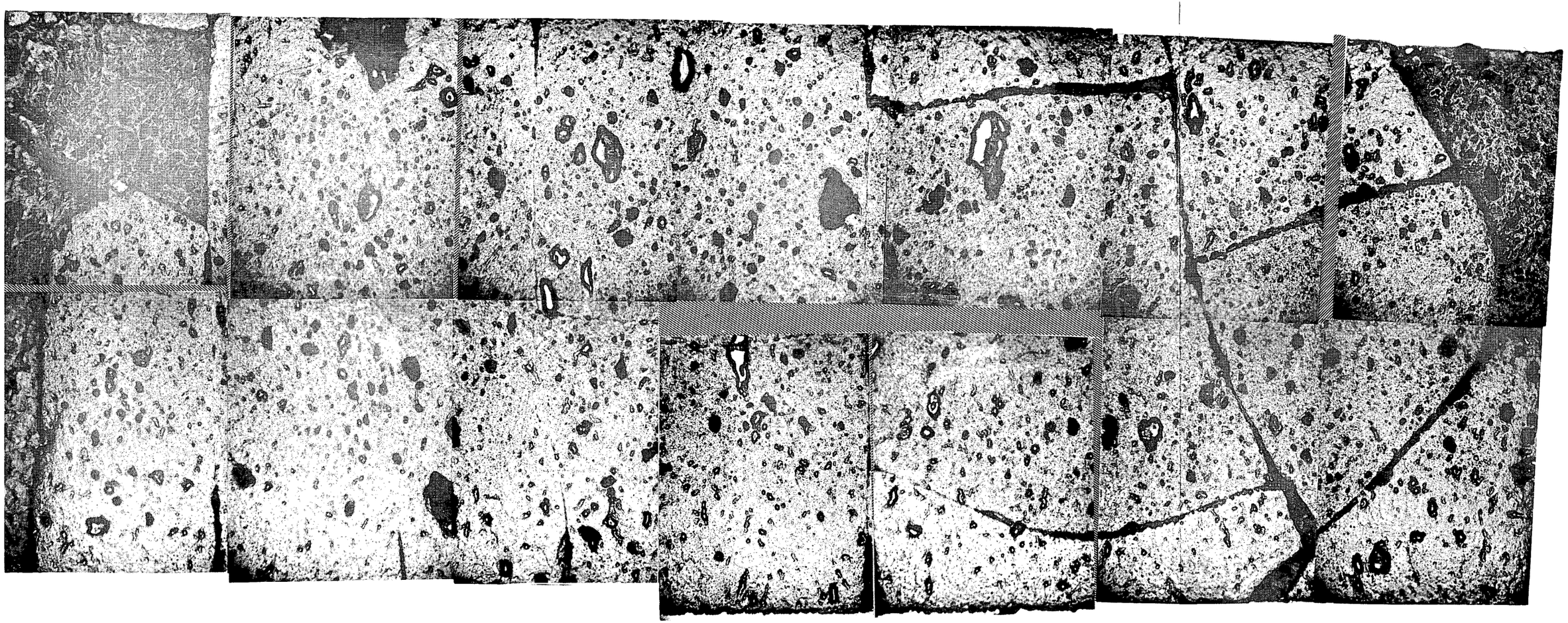
80 wt% UO_2-BeO, Unirradiated and etched

FIGURE 13 11.5-63-177

Typical appearance near outer surface. Note the cracks, and void distribution. The large voids were probably formed during fabrication.

FIGURE 11. PHOTOMICROGRAPH OF 80 WT % UO_2-BeO SPECIMEN 13

NOTE: AREAS SHOWN ////// WERE NOT COVERED BY THE PHOTOMICROGRAPHS



11.5-63-059

FIGURE 12. LONGITUDINAL SECTION THROUGH 70 WT % UO_2-BeO FUEL SPECIMEN

been caused by agglomeration of fission gas voids, although agglomeration of the original void volume in this material may have contributed to the post-irradiation appearance of the particles. Also of interest was the change in UO_2 particles near pellet surfaces from an irregular but distinct particle outline to an indistinct outline (see Figure 14). The reaction of these particles toward etchants had changed considerably and it was difficult after irradiation to delineate the microstructure.

Porosity of fuel particles located more centrally in the 70 wt% UO_2 did not appear greatly altered from the pre-irradiation appearance (Figure 15 shows typical views of central pellet structures). It can be observed that while the particle edges were more regular than in unirradiated specimens, the UO_2-BeO interface was more sharply defined than that noted around particles close to the pellet surface. Particle microstructures could be resolved more definitely by etching in this more central location. The large voids which appear at the UO_2-BeO interface were not observed in pre-irradiation views. It is possible that these were gas voids or a very brittle zone caused by fission fragment bombardment, which pulled out during specimen preparation. Around the edges of the smaller fuel particles a zone can be noted which might also have been caused by fission fragment damage.

The differences noted between particles located at edge and center of fuel pellets may be attributed to the large differences in burnup between the two locations. As is indicated in Figure 13 in a specimen which was exposed to an average burnup of 6.4×10^{20} fissions/cc, it was estimated that particles on the pellet surface received burnups of 30×10^{20} fissions/cc.

Microstructures of the 80 wt% UO_2 bodies did not show the edge-center differences observed in 70 wt% UO_2 bodies (Figure 13 displays a pre-irradiation view of this fuel composition and Figure 16 shows post-irradiation views). It appears that some particle agglomeration and a change in the BeO-UO_2 relation had occurred. It appears that the BeO phase had been interrupted with the UO_2 phase becoming dominant.

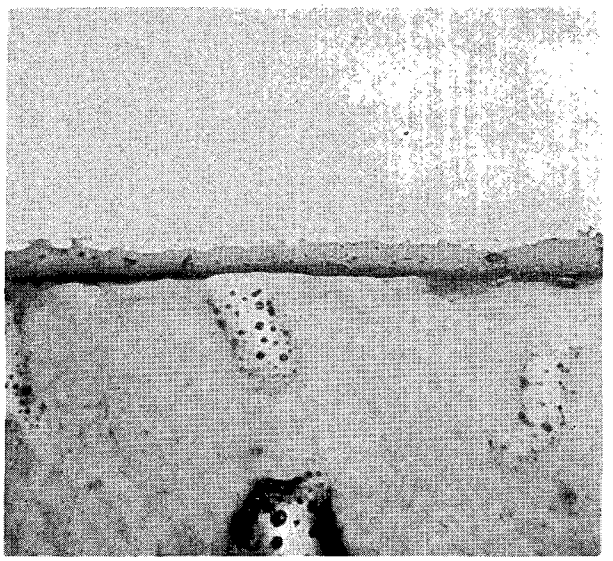
Large elongated voids were found within the fuel pellets in specimen 13. These appeared to have been caused by defects introduced during pellet fabrication rather than melting (Figure 17).

The Hastelloy X cladding did not appear adversely affected by the exposure. A curious formation observed (Figure 14) appeared to indicate evidence of a reaction between the cladding and fuel. Examination of the cladding as etched, however, did not support this indication, since no reaction could be detected in the cladding microstructure. There was no evidence of the grain boundary precipitates which were observed in an earlier experiment where solid UO_2 contained in Hastelloy X was the investigated (Ref. 1).

H. X-RAY DIFFRACTION

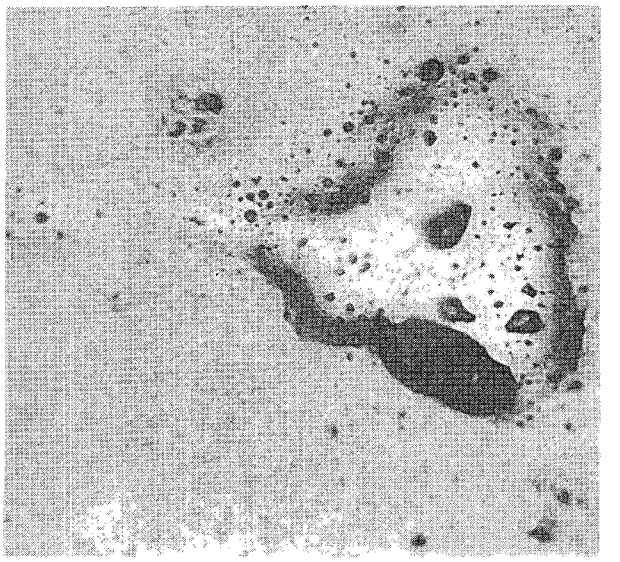
Selected cross-sectioned samples from specimen 6 (70 wt% UO_2-BeO) and specimen 11 (80 wt% UO_2-BeO) were removed, ground into a fine powder and mounted for X-ray diffraction studies. An unirradiated 70 wt% UO_2-BeO specimen was prepared in a similar manner and the results of X-ray diffraction studies compared with those of the irradiated samples. The results of X-ray diffraction

CONDITION OF UO_2 PARTICLES NEAR OUTER EDGE OF FUEL PELLETS (BeO-70 WT % UO_2 FROM CAPSULE MTR GCR-3. NOTE THE VOIDS IN THE UO_2 PARTICLES BUT ARE LOCATED AT THE UO_2-BeO INTERFACE



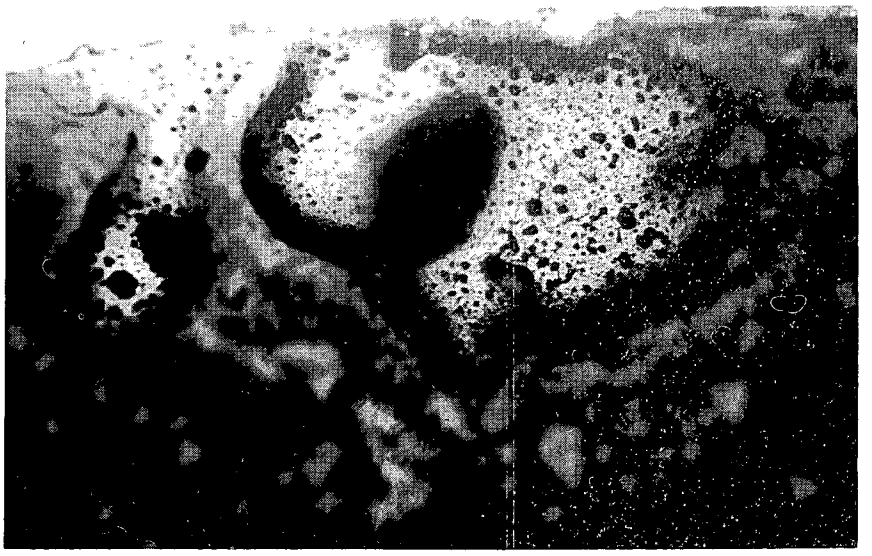
AS-POLISHED 250X

(a) Specimen 6 irradiated to a burnup of 6.4×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F. Burnup of fuel particle near pellet surface estimated to be about 30×10^{20} fissions per cm^3 of UO_2.



AS-POLISHED 250X

(b) Specimen 4 irradiated to a burnup of 6.3×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F.



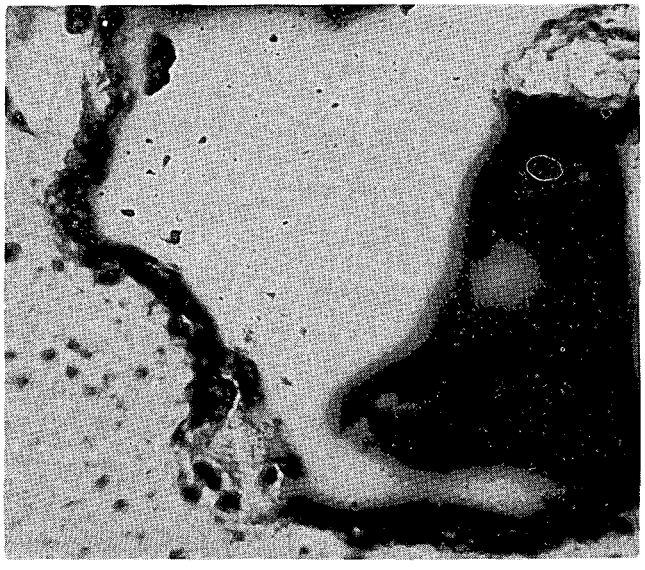
AS-POLISHED 250X

(c) Specimen 6 irradiated to a burnup of 6.4×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F.

FIGURE 14

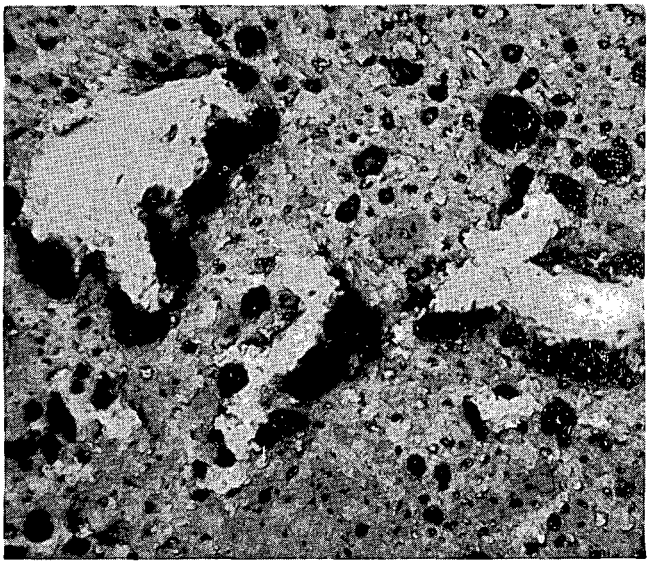
11.5-63-060

CONDITION OF UO_2 PARTICLES NEAR CENTER OF FUEL PELLETS (BeO-70 WT % UO_2) FROM CAPSULE MTR GCR-3. NOTE VOIDS ARE NOT WIDENT IN THE UO_2 PARTICLES BUT ARE LOCATED AT THE UO_2-BeO INTERFACE



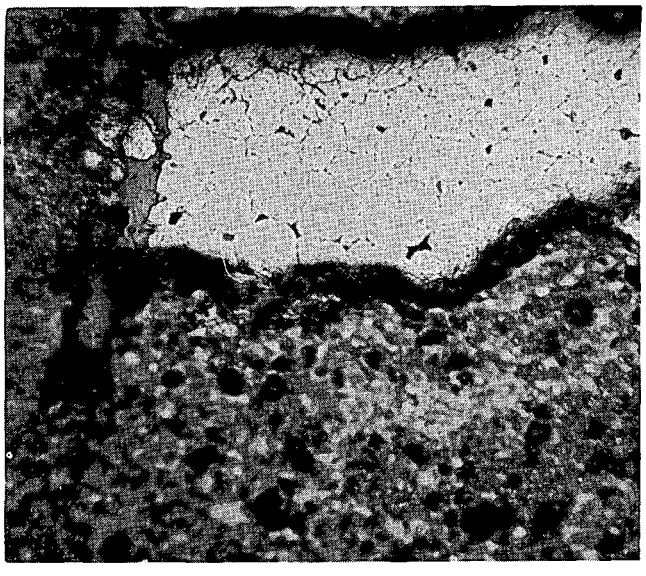
AS-POLISHED 250X

(a) Specimen 6 irradiated to a burnup of 6.4×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F



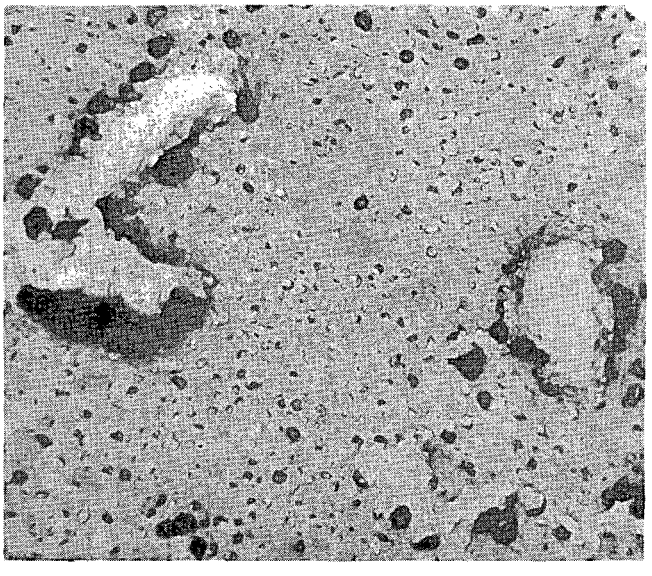
AS-POLISHED 250X

(b) Specimen 4 irradiated to a burnup of 6.3×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F. Note voids at UO_2-BeO interface



ETCHED 250X

(c) Specimen 6 irradiated to a burnup of 6.4×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F



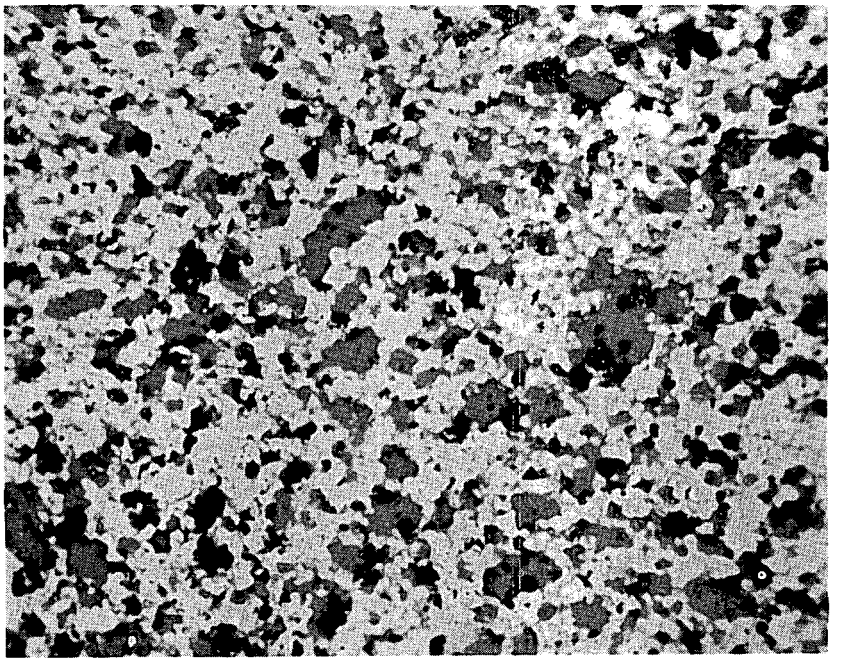
AS-POLISHED 250X

(d) Damaged portion of Specimen 4 irradiated to a burnup of 6.3×10^{20} fissions/cc of pellet at a maximum clad surface temperature of 1600°F

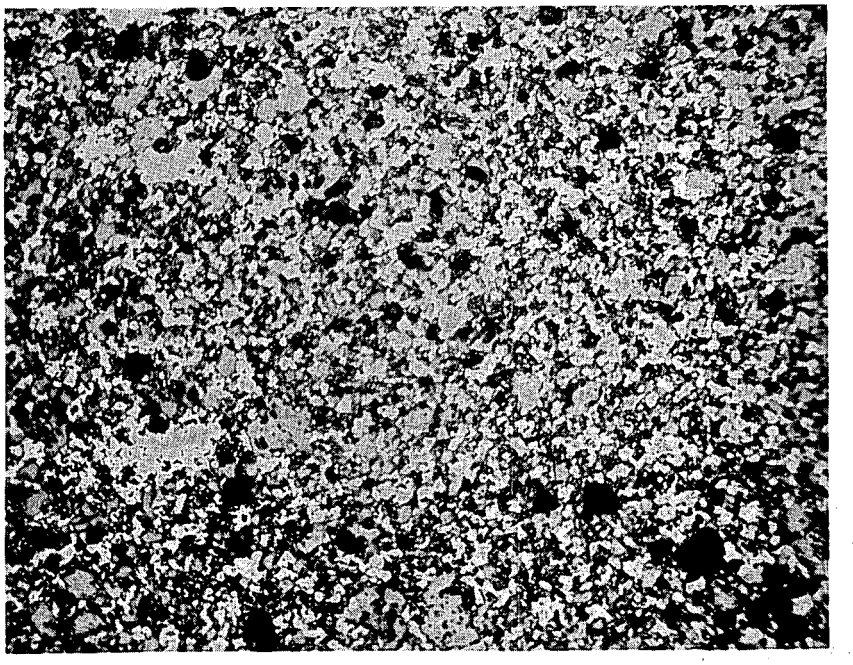
11.5-63-061

FIGURE 15

SPECIMEN 13 (BeO-80 WT % UO_2) IRRADIATED TO A BURNUP OF 5.9×10^{20} FISSIONS/cc OF PELLET AT A MAXIMUM CLAD SURFACE TEMPERATURE OF 1580°F



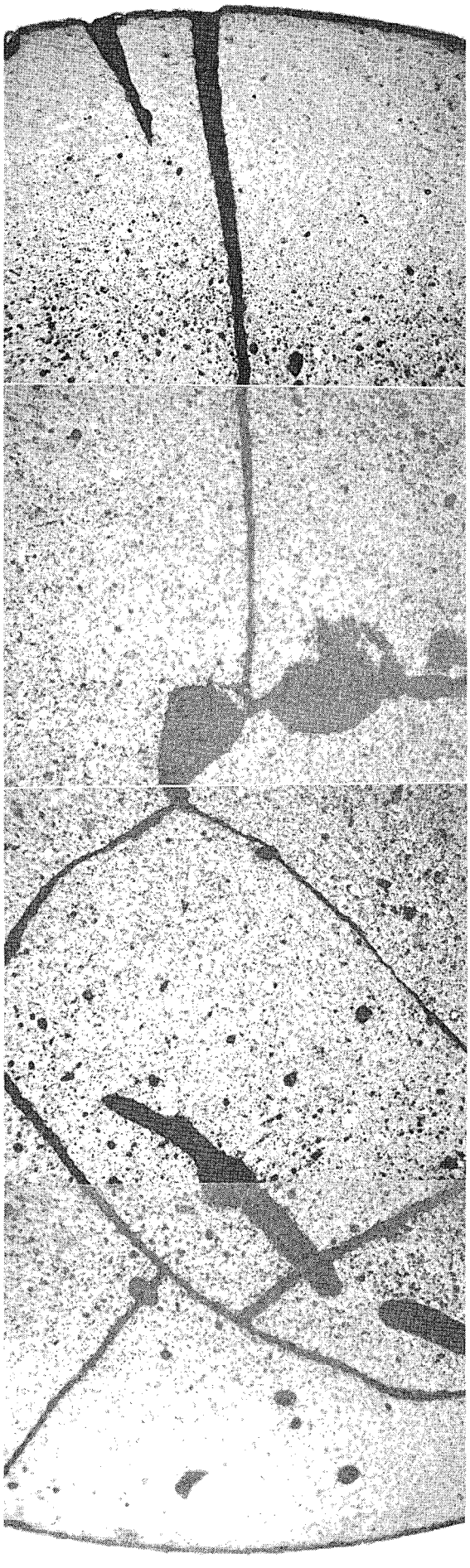
AS-POLISHED 500X



ETCHED 250X

FIGURE 16 11.5-63-062

METALLOGRAPHS OF SPECIMEN 13



The metallographs of specimen 13 (80 wt % UO_2-BeO) illustrates the condition of the specimen. These voids probably orginated during fabrication of the specimen rather than as a result of irradiation

35X

FIGURE 17

measurements on specimen 6 (70 wt% UO_2-BeO) indicated an average UO_2 crystal contraction of 0.26% and an internal strain in the UO_2 crystal of 0.18%. It should be noted that these data refer to average conditions in the fuel pellet; however, presence of the large burnup gradient existing between the edge and center of these pellets may not be reflected by these results.

The X-ray diffraction data were obtained with a shielded diffractometer incorporating a crystal-monochromated detector utilizing electronic discrimination. An increased broadening of the UO_2 diffraction lines, (111), (220), and (311) was observed. The broadening of these lines was interpreted to yield the microstresses reported above. No X-ray diffraction pattern for BeO could be obtained from these specimens. It was believed that this difficulty was due to the high UO_2 loading and the greater sensitivity of a diffractometer for UO_2 than BeO. Special treatment of these specimens was required to obtain a diffraction pattern of the irradiated BeO. Specimen 7 was entirely consumed in previous experiments so that specimen 3 (70 wt% UO_2-BeO), which had received an average burnup of 5.2×10^{20} fission/cc, was used in experiments to determine the BeO patterns. In preparing these specimens, the UO_2 was extracted with nitric acid and the BeO remaining was collected. The positions and breadths of BeO X-ray diffraction lines (100), (002) and (101) and (110) were then measured. It was apparent from the results that there was a small increase in breadth of the diffraction lines of the irradiated BeO which was interpreted as lattice strain of 0.044%. Except for this small effect, there was essentially no change in the irradiated BeO contained in specimen 3. Apparently, the volume of BeO located within recoil range of the UO_2 particles, which should have suffered severe lattice damage, was not sufficiently great to affect the overall results of this study (Figure 14). Results of the X-ray diffraction patterns of UO_2 from 6 and 11 and an unirradiated sample are shown in Figure 18. Patterns of irradiated and unirradiated BeO are shown in Figure 19.

FIGURE 18. X-RAY DIFFRACTION PATTERNS OF UO_2 FROM SPECIMENS 6 AND 11, CAPSULE MTR GCR-3

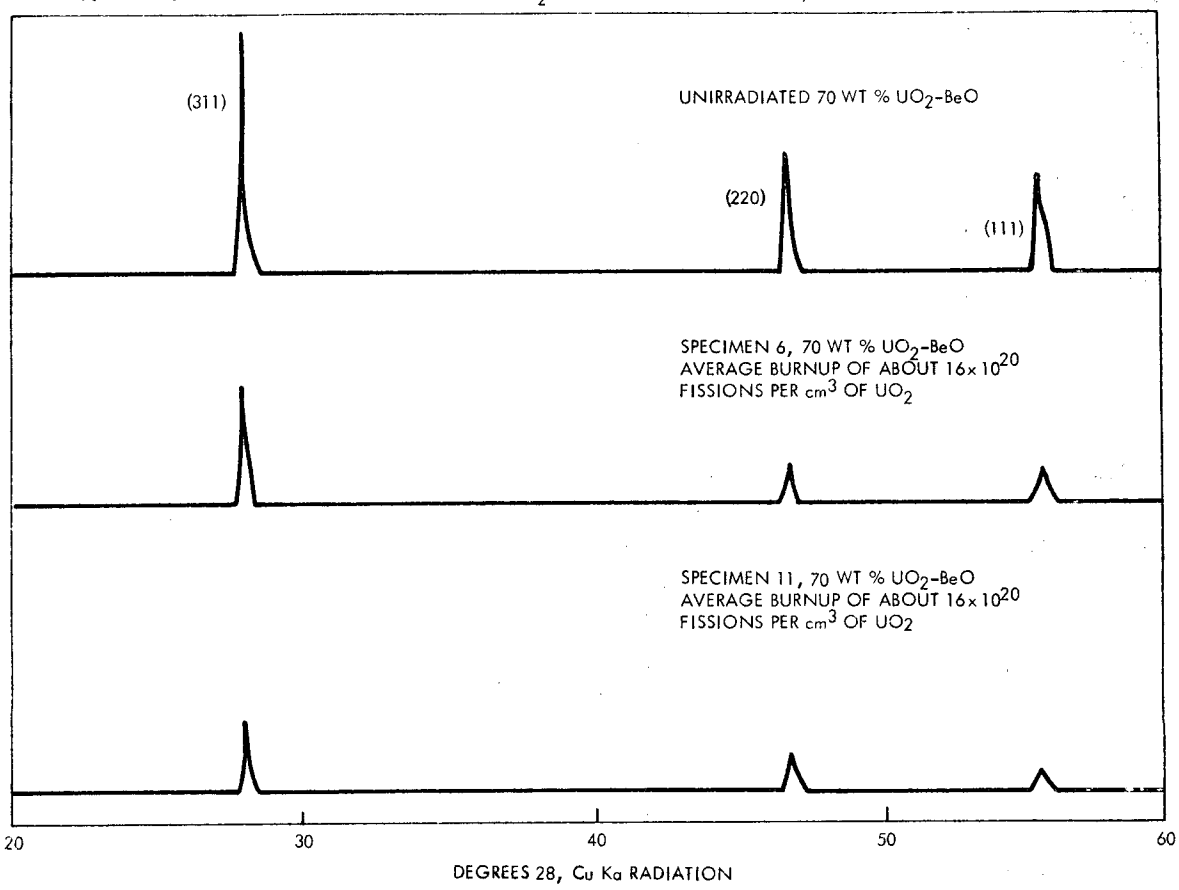
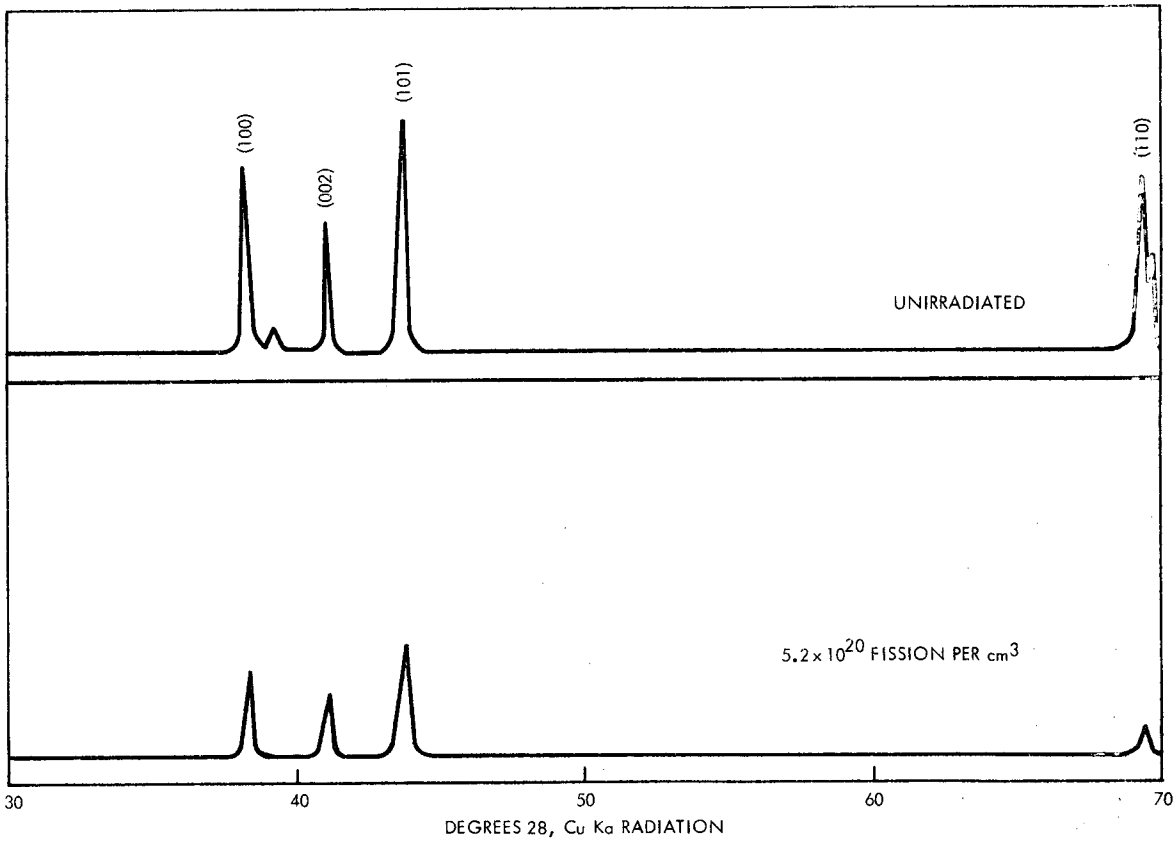


FIGURE 19. X-RAY DIFFRACTION PATTERNS OF BeO FROM SPECIMEN 3 CAPSULE MTR GCR-3



115-63-063

IV. DISCUSSION

On the basis of visual, dimensional and microscopic evaluation, the UO_2-BeO fuel withstood the described exposure with few apparent detrimental effects. Metallographic examination revealed that there was, in fact, damage to the BeO immediately surrounding the UO_2 particles; that specimen 11 had failed by what appeared to be central core melting, and that UO_2 particles located close to the pellet surface had been severely damaged or destroyed. No other obvious effects were noted in either the metallographic or visual examinations. A comparison of pre- and post-irradiation dimensional and density measurements did not indicate that significant swelling had occurred as a result of this irradiation in any but specimen 11.

The results of thermocouple measurements and heat generation calculations indicate that specimen cladding surface temperatures measured ranged between 1300° and 1650°F. The average fission burnup of U-235 in the specimen exposed to the highest neutron flux was about 8.5 atom%, while a specimen exposed to the lowest flux was determined as 4.8 atom%. Macro examination of the fuel showed the presence of predominately radial cracking, although a tendency toward circumferential cracking was observed.

The release of gaseous fission products generated in the fuel, (again except for specimen 11 which had experienced central melting, swelled and ruptured its cladding) was quite small. It was assumed that all the fission gas detected within the capsule cavity was released from specimen 11 since only this specimen had a cladding failure.

There is no obvious explanation for the central core void formed in the fuel pellets of specimen 11. Surface temperatures measured on this particular specimen during the early irradiation cycles did not indicate surface temperatures high enough to support central core melting. Unfortunately, all thermocouples failed after the first two irradiation cycles so that there was no accurate surface temperature data after this time. Data based upon gamma scanning of the specimens indicated specimen 13, also of 80 wt% UO_2-BeO, to have been in a higher average temperature than specimen 11, yet there was no evidence of melting in specimen 13. However, specimen thermocouples, when operative, measured considerably higher cladding temperature on specimen 11 than on specimen 13.

Central fuel temperatures were estimated for two cases to investigate the cause of failure: In one case the fuel pellet was centered, in the other case it rested against the tubing wall at one point. Pure UO_2 and BeO-UO_2 fuels were investigated under capsule conditions for comparison. The results of this study, tabulated in Table 8, indicate relatively high central temperatures in the UO_2 fuel. The BeO-UO_2 fuels showed quite low central temperatures, considerably lower than that required to cause central melting.

TABLE 8

TEMPERATURE DISTRIBUTION IN FUEL SPECIMENS OF UO_2 AND BeO-UO_2 CAPSULES

	Fuel Central Temp. °F	Edge of Fuel Pellet °F	Cladding Temp °F
UO_2 Fuel[(a)]	3925°F	2290°F	1775°F
UO_2 Fuel[(b)]	3856°F	2552[(c)]	
80 wt% UO_2-BeO	2950	2280	1685
70 wt% UO_2-BeO	2610	2060	1570

(a) Pellet centered

(b) Pellet in contact with cladding

(c) Side not in contact with cladding

A comparison of melting point vs composition for a range of UO_2-BeO compositions (Figure 20) supports the contention that the estimated UO_2-BeO central temperatures are too low to result in melting. If the internal structure of this mixture had changed in such a way that UO_2 became the matrix rather than dispersed phase, however, the effective thermal conductivity of the mixture would be greatly reduced. The conductivity of the body could become more comparable to that of UO_2, where the calculated temperature was of sufficient magnitude to cause melting in a diluted system. The post irradiation metallography supports this contention to a great extent; as shows in Figures 10 and 16. It appears from Figures 10 and 16 that there has been an agglomeration and stratification of UO_2 to form a continuous phase.

The 70 wt% UO_2-BeO fuel melting point is sufficiently greater than the 80 wt% to preclude melting with a 70 wt% UO_2-BeO composition at the central temperatures estimated (Figure 20).

Metallographic examination of fuel from the 70 wt% UO_2-BeO specimens revealed a brittle or easily etched zone in the BeO immediately adjacent to the UO_2 particles. This condition can be observed in particles shown in Figures 14 and 15. This condition has apparently been caused by fission fragment damage to the BeO lattice in these areas. It was not possible to detect the damage by X-ray diffraction as it is confined to an extremely localized area and constitutes a very small proportion of the total volume present.

MELTING POINT VERSUS COMPOSITION FOR UO_2 - BeO

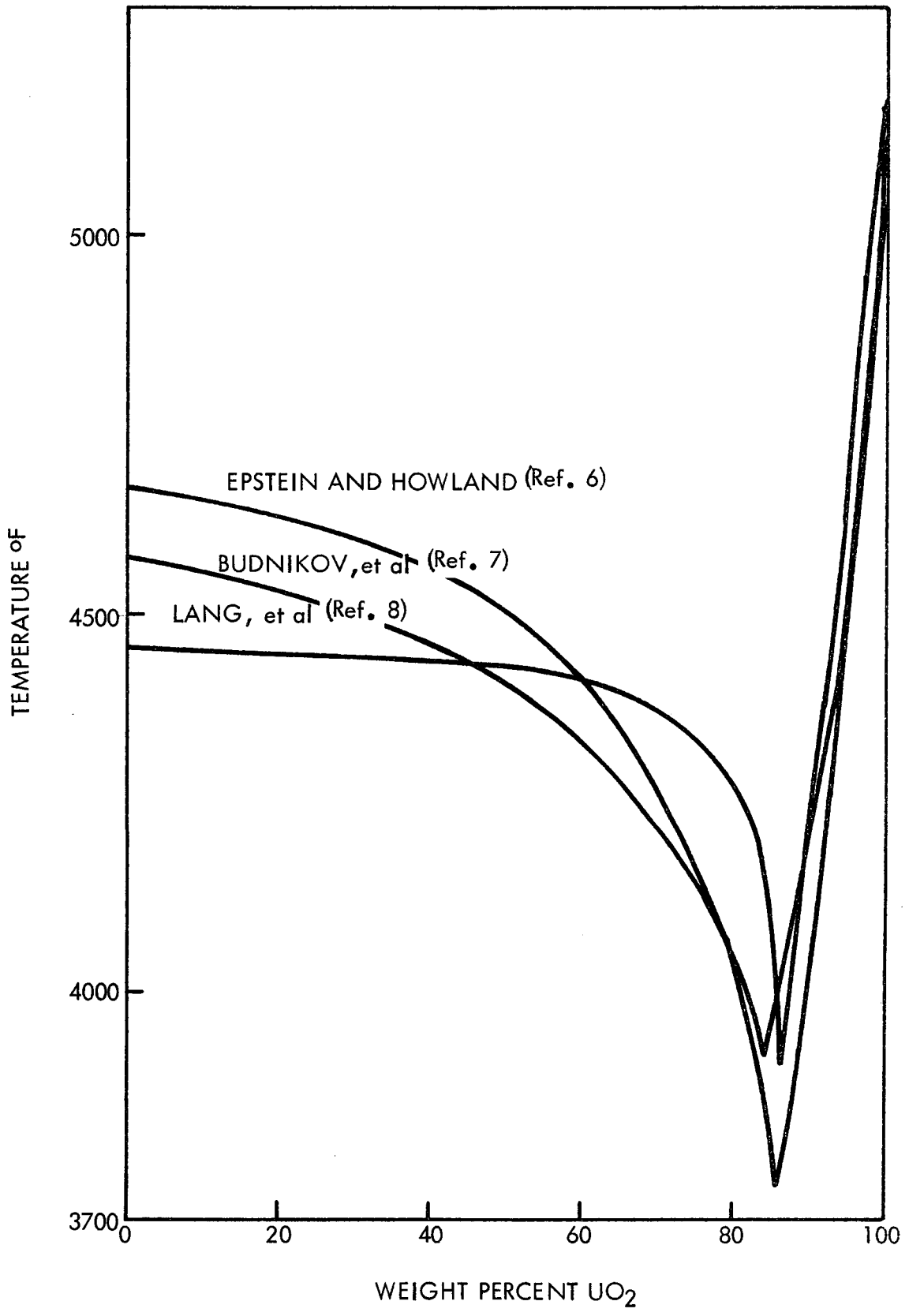


FIGURE 20

11-5-63-290

Electron microprobe analysis might have detected these changes by virtue of the capability of this equipment to evaluate specific points in a specimen. Unfortunately, radiation-shielded equipment was not available so that this point could be resolved.

A marked difference in radiation damage between the edge and center of fuel pellets was described earlier. Particles of UO_2 located close to the pellet edge were observed to contain many large spherical voids. This is a quality usually attributed to high burnups in dispersion type fuels and has obviously occurred in this case. The voids probably resulted from accumulation of fission product gases. Fuel particles more centrally located were observed to be relatively free from these voids. It was observed that large voids were located at the interface between the BeO matrix and UO_2 particles near the fuel center. These may indicate that the fission gases were diffusing from the UO_2 particles and lodging in the adjacent BeO where agglomeration occurred because of higher fuel temperatures in the fuel center.

Another point mentioned earlier concerned the indistinct appearance of the UO_2-BeO interface and the difficulty encountered in resolving the UO_2 microstructure in particles close to the fuel pellet surface. This phenomenon has been attributed to severe damage or complete destruction of the UO_2 lattice in this particular region. Here again, as in the instance of the BeO described earlier, this damage could not be detected by diffraction as the total amount of UO_2 which received an exposure sufficient for lattice destruction was quite small.

Damage resulting in complete destruction of the BeO and UO_2 lattice has been described by Bleiberg (Ref. 9) and Berman (Ref. 10). They reported exposure levels of 18×10^{20} fissions/cc of UO_2 were required to produce measurable destruction of crystal lattice. Since that time, however, further studies with similar materials have shown that complete lattice destruction did not occur. Instead, very fine grains were formed which gave the appearance of an amorphous structure with the equipment and techniques then available*. Temperatures also effect the amount of damage. Studies performed by Shields (Ref. 11) indicated that damage to BeO from neutron exposure is reduced as irradiation temperatures are increased to temperatures in the 1400°F range.

In view of the foregoing points, it is not unexpected, then, that neither the UO_2 nor the BeO in this fuel were extensively damaged.

*Personal communication to J. H. Saling, BMI from Thomas R. Padden, Westinghouse-Bettis Atomic Power Laboratory, 17 January 1963.

CONCLUSIONS:

The following conclusions can be drawn from this fuel irradiation experiment:

1) With the exception of one specimen, negligible changes occurred in the dimensional properties of the fuel irradiated in this experiment to average burnups ranging from 5.0×10^{20} to 6.4×10^{20} fissions/cc of fuel core at specimen clad surface temperatures ranging from 1300° to 1710°F.

2) Under the conditions of this experiment, the only changes observed in the microstructure of the UO_2 or BeO was the appearance of fission gas bubbles in the UO_2 particles near the outer periphery of the fuel; an apparent fission fragment damage zone in the BeO at the interface between the UO_2 and BeO; and the apparent destruction of the microstructure of UO_2 particles located near the surface of the fuel.

3) Fission gas loss from the fuel was very low (0.6 to 2.7%) for all specimens with the exception of specimen 11 which released about 69% of the total formed in the fuel.

4) X-ray diffraction data indicated little change in the crystal structure of either the BeO or UO_2 as a result of irradiation. However, these data were affected by the small volume of BeO damaged by fission fragments as compared to the total volume of BeO, and by the small fraction of the total quantity of UO_2 particles which was subjected to burnups of sufficient magnitude to destroy the lattices. Essentially the X-ray diffraction data represented average conditions on the fuel.

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LEGAL NOTICE

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The information herein is regarded as preliminary and subject to further checking, verification and analysis.