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POSTIRRADIATION EXAMINATION OF HIGH BURNUP EGCR PROTOTYPE
FUEL CAPSULES

M. F. Osborne
E. L. Long, Jr.
J. G. Morgan

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REACTOR CHEMISTRY DIVISION
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METALS AND CERAMICS DIVISION

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POSTIRRADIATION EXAMINATION OF HIGH BURNUP EGCR PROTOTYPE
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ABSTRACT

We present here the results of the examination of nine fuel capsules which were irradiated in the Engineering Test Reactor (ETR) for periods of three to four years. The irradiation capsules were prototypes of the fuel elements for the Experimental Gas-Cooled Reactor (EGCR), the construction of which has been discontinued. Some of the capsules were examined much more thoroughly than others. Only one of this group of nine capsules was found to have suffered a loss of cladding integrity. Three stainless steel containment vessels which had been exposed to nitrogen at high temperatures were examined; two of these vessels had experienced appreciable nitriding.

INTRODUCTION

A large number of fuel capsules which simulated the Experimental Gas-Cooled Reactor (EGCR) fuel rods except for length have been irradiated in the Oak Ridge Research Reactor (ORR) and the Engineering Test Reactor (ETR). The results of most of these capsules have been reported.^{1,2,3} In general, the capsules discussed in this report were examined in less detail than were those capsules reported previously.

These prototype EGCR fuel capsules were composed of sintered UO_2 pellets contained in stainless steel tubing. All of the fuel capsules were of the same size and geometry, but such parameters as fuel fabrication techniques, cored vs solid fuel pellets, thermocouple locations, operating temperature of the cladding, and fuel burnup were studied. We examined the capsules for dimensional stability, cladding integrity, axial radioactivity distribution, and changes in the microstructure of the fuel and cladding.

Many people from several ORNL Divisions participated in this work. The fuel capsules were fabricated and the experiments were designed and irradiated by members of the Metals and Ceramics Division and the Reactor Division, respectively. Personnel of Phillips Petroleum Company at the NRTS assisted with the ETR irradiations. Reactor Chemistry Division personnel were responsible for postirradiation examination and the interpretation of results.

CAPSULE CONSTRUCTION

The evaluation of the EGCR fuel element design⁴ evolved through several stages of irradiation capsules; those discussed in this report represent the earliest stages of construction.

Type 304 stainless steel was used for cladding all capsules.^{5,6} Prior to use the tubing (nominal inside diameter of 0.711 in. and a wall thickness of 0.020 in.) was carefully inspected by visual, dye penetrant, dimensional, ultrasonic, and radiographic methods. Samples of the tubing and end cap material were analyzed chemically, and their mechanical properties (tensile strength, yield strength, and elongation) were determined.

All of the capsules were fueled with sintered, high density UO_2 pellets, which were nominally 0.705 in. OD and 0.5 in. long. Four capsules contained solid fuel pellets, three contained pellets with a 0.323-in.-diameter hole, and two contained similar cored pellets with a BeO bushing inserted in the hole (see Table 1). The fuel enrichment was 0.71% (normal) for the solid pellets and about 1.0% for the cored pellets (see Fig. 1). The capsules contained either a six-inch-long column of cored fuel pellets or a five-inch-long column of solid fuel pellets; hollow stainless steel bushings were placed at each end of the fuel column in the latter case to provide gas expansion volume. The UO_2 used in most of these capsules did not meet EGCR specifications because it contained excess carbon, nitrogen, and oxygen. Magnesium oxide pellets were placed at each end of the fuel column to insulate the UO_2 from the stainless steel end caps.

Table 1. Properties of UO_2 Fuel Pellets

Capsule No.	Pellet Geometry	Enrichment (% ^{235}U)	Avg. Bulk Density (g/cc)	O/U Ratio	Impurities	
					Nitrogen (ppm)	Carbon (ppm)
E-2	Solid	0.712	10.47	2.003	300	500
E-3	Solid	0.712	10.46	2.003	300	500
E-5	Cored	1.06	10.37	2.038	1500	200
E-10	Cored-BeO	1.03	10.51	2.019	500	40
E-11	Cored-BeO	0.995	10.43	2.005	5	120
E-1R	Solid	0.712	10.46	2.003	300	500
E-4R	Solid	0.712	10.45	2.003	300	500
E-5R	Cored	0.962	10.29	2.002	13	82
E-12R	Cored	0.995	10.42	2.005	5	120

All metal components were cleaned with acetone immediately prior to assembly. The top end caps were press-fitted into the cladding and Heliarc welded in place in a helium-filled dry box. The completed capsules were thoroughly inspected,⁷ and those with visible surface defects or with leak rates greater than 1×10^{-8} cm³/sec were rejected. The external dimensions (diameters, profiles, and lengths) of each capsule were recorded for comparison with the corresponding dimensions after irradiation.

IRRADIATION CONDITIONS

The capsules were irradiated in the assembly⁸ shown in Fig. 2. Six stainless steel sheathed Chromel-Alumel thermocouples were strapped to the capsule with 0.005-in.-thick stainless steel foil; the strap also served as a thermal neutron flux monitor. The capsules were immersed in NaK, which acted as a relatively isothermal heat transfer medium. Temperature was controlled by varying the composition of the gas mixture (helium and nitrogen or helium and argon) in the annulus between the NaK vessel and the outer container.

Fig. 1. Construction Details of ETR-Irradiated Fuel Capsules. Some capsules contained solid fuel pellets, and some capsules had BeO rods inserted in the center hole of the hollow fuel pellets.

Fig. 2. Experimental Assembly for Irradiation of EGCR Prototype Capsules in ETR.

Except for irradiation time, operating conditions for all of these experiments were similar. These data are shown in Table 2. The maximum design values for power and heat flux were 35,000 Btu/hr·ft and 190,000 Btu/hr·ft², respectively. However, operational data indicated that the actual power levels departed from design values by varying and indefinite amounts. Cladding temperatures were designed to be either 1300°F avg or 1600°F max., and were lower than those values during most of the operating periods. All of the capsules were irradiated under an external pressure of 300 psia, which tended to collapse the cladding against the fuel.

Table 2. Irradiation Data

Capsule Number	Cladding Temp.		Irradiation Time (Full Power Days)	Avg. Thermal Neutron Flux (n/cm ² ·sec)	Burnup ^a (MWD/MT UO ₂)	Maximum Power ^b (% of Design)
	Design (°F)	Actual (°F)				
E-2	1300	1300	657	2.01 x 10 ¹³	4500	61
E-3	1600	1500	657	3.37 x 10 ¹³	5790	111
E-5	1300	1250	704	2.76 x 10 ¹³	9500	111
E-10	1600	1500	657	2.55 x 10 ¹³	7400	110
E-11	1300	1300	742	2.59 x 10 ¹³	6300	87
E-1R	1600	1550	568	3.33 x 10 ¹³	6500	112
E-4R	1300	1250	568	3.68 x 10 ¹³	7500	97
E-5R	1600	1500	672	3.38 x 10 ¹³	7300	117
E-12R	1600	1450	542	4.38 x 10 ¹³	7600	98

^aCalculated from neutron flux data.

^bFor any one cycle, as determined from operating data. (Design power was 35,000 Btu/hr·linear ft.)

Radioactivity detectors were attached to the helium cover gas system, which maintained the external capsule pressure. This technique of detecting capsule failures was not consistently dependable, either because the detectors were not sensitive enough or because the signals were not properly interpreted by the operator.

POSTIRRADIATION EXAMINATION

After irradiation each capsule was removed from the NaK container and the excess NaK was rinsed off in light mineral oil. A sample of the NaK was analyzed for ^{137}Cs , since its presence would indicate a cladding failure. The stainless steel thermocouple strap and the sheathed thermocouples were then removed to permit thorough inspection of the cladding. One or more sections of the thermocouple strap were analyzed for ^{60}Co , and the resulting data were used to calculate the thermal neutron dose at the exterior of the capsule. Capsule E-3, which had a visible cladding fracture, was immersed in iso-butyl alcohol to react the NaK which had reached the interior of the capsule. (The combination of thermal cycling and pressurization of the cover gas forced the NaK through the fractures into the capsule.) After the capsules were photographed and checked for leaks, the external dimensions were measured, and gamma radioactivity vs length (called a gamma scan) was recorded. The gamma scan curves showed fuel location and the fuel burnup profile. Three of these capsules were disassembled for detailed examination of the cladding and fuel, and the other six capsules were transferred to another program for use in fission products behavior studies. In addition, the NaK tubes from three of these experiments were examined for the extent to nitriding of the outside surface, which had been exposed to the helium-nitrogen control gas mixture during irradiation.

Capsule Appearance

The general appearance of seven of the capsules was normal; Fig. 3, showing capsule E-2, is representative. These capsules had diameter changes of up to about 0.010 in., and several had small circumferential ridges at pellet interfaces. Capsule E-12R was the most severely ridged of any EGCR prototype capsule examined. (See Fig. 4) The cladding surface had a roughened, granular appearance also, and diameter measurements showed the capsule to be slightly swollen. (In most capsules which operated at 1600°F, the cladding had collapsed against the fuel.)

Capsule E-3 could not be withdrawn from the NaK tube in the usual manner. The tube was removed by slitting it into two 180° sections. The sheathed thermocouples and the thermocouple-retaining strap were

R-17826

Fig. 3. Capsule E-2, Which Illustrates Typical Appearance of EGCR Prototype Capsules. 0.6X.

Fig. 4. Capsule E-12R, Which Experienced Extensive Cladding Deformation but Did Not Fail.

found to be welded to both the capsule and to the NaK tube, explaining the disassembly difficulties. Two large cladding fractures were apparent near the mid-length of the capsule; the larger fracture was approximately one in. long by 1/8 in. wide, as shown in Fig. 5. The cladding surface near the fractures had an unusual granular appearance.

NaK Radioactivity

Gross fission product radioactivity (^{137}Cs) was found in the NaK from capsule E-3, and a very small amount was found in the NaK from capsule E-1R, but no other indication of failure was found. The absence of such radioactivity in the NaK from all of the other capsules indicated that there were no other failures.

Gamma Scans

Four of the capsules were gamma scanned; only capsule E-12R showed an unusual distribution of radioactivity. The radioactivity gradient along the length of capsule E-12R indicated that the neutron flux had been about 30% greater at the bottom end of the capsule than at the top end. This effect is illustrated in Fig. 6, in which we compare the gamma scan of capsule E-12R with that from capsule E-3, which had a typical radioactivity distribution.

Dimensional Measurements

Dimensional data were obtained for six of the capsules, as summarized in Table 3. With the exception of capsule E-3, which was severely swollen, we found no unusually large dimensional changes. The cladding of capsules E-10 and E-11 had collapsed against the fuel, as evidenced by small diameter decreases. Small diameter increases (~ 0.010 in.) were found in capsules E-1R and E-12R. In the latter case, the extensive wrinkling of the cladding made accurate measurements difficult and probably contributed to the observed diameter increases. The average diameter of capsule E-3 in the vicinity of the cladding fractures had increased as much as 0.063 in. In addition, this capsule was the only one which had experienced a significant change in length, an increase of about 0.10 in.

R-24882

R-24508

(a)

(b)

Fig. 5. Capsule E-3, Which Suffered Cladding Failure. (a) Overall view of the capsule, 0.3X. (b) Fracture region. Note thermocouples and foil which were welded to the cladding. 2.2X.

Fig. 6a. Gamma Scan of Capsule E-12R. Note the axial radioactivity gradient, indicating ~ 30% higher burnup at the bottom end than at the top end of the capsule.

Capsule E-3

Fig. 6b. Gamma Scan of Capsule E-3. The radioactivity distribution is typical of EGCR capsules. Note the flux peaking at each end of the fuel column.

Table 3. Diameter Changes for ETR-Irradiated EGCR
Prototype Capsules

Position (in. from bottom end)	Change in Average Diameter, mils					
	E-3	E-10	E-11	E-1R	E-12R	E-4R
0	--	--	--	--	--	--
0.5	+0.1	--	-0.6	--	-4.1	--
1.0	+0.5	-2.8	-4.4	+10.6	+0.3	+0.1
1.5	+15.5	--	-3.8	--	-0.6	--
2.0	+33.5	-4.4	+6.8	+7.2	+1.6	-0.3
2.5	+62.9	--	+7.7	--	+1.0	--
3.0	+40.5	-5.3	-3.7	+11.4	+6.8	-0.8
3.5	+39.1	--	-2.1	--	+6.7	--
4.0	+25.7	-2.7	-4.3	+10.8	+4.9	-0.5
4.5	+24.9	--	-4.8	--	+5.8	--
5.0	+24.4	-1.7	-3.9	+4.9	+5.1	0.0
5.5	+17.7	--	-3.0	--	+8.6	--
6.0	+1.8	-2.0	-0.4	+2.5	+7.2	+0.7
6.5	-3.9	-0.2	+0.6	-5.9	+5.6	+2.0

Fuel Appearance

Except for capsule E-3, the macroscopic appearance of the UO_2 was normal in all capsules that were examined in detail. Most of the pellets were broken radially into four to ten pieces, but no severe fracturing or powdering was found. Circumferential fracturing had occurred in the solid fuel pellets of capsule E-3 at a radius of about 0.25 in. (Fig. 7). Although columnar grains appeared to exist in the central region, we found no central void.

R-26651

Fig. 7. Transverse Section from Capsule E-3 Which was Examined Metallographically. Note the radial and circumferential fracturing in the UO_2 and the cladding fracture and thermocouple welded to cladding. 100X, Etched.

Metallographic Examination

A transverse section of fuel and cladding was selected from the mid-length region of each capsule for microstructural study. The section from capsule E-3, which is shown in Fig. 7, included the cladding fracture and the sheathed thermocouple which had been welded to the cladding during irradiation. The shapes of the fracture surfaces, shown in Fig. 8, indicated a ductile, cup-cone type fracture. The ferritic layer on the outside surface of the cladding was much thicker (~ 0.010 in.) near the fracture than in other regions (average ~ 0.001 in.). In addition, it should be noted that the ferritic layer extended well into the fractured surfaces. The appearance of the UO_2 in the central and outside diameter regions (i.e., maximum and minimum temperature regions) are compared in Fig. 9. The elongated grains in the central region indicated that this part of the fuel reached the temperature of columnar grain growth, which is a minimum of 3000°F , although no central void was found. A few small chips of UO_2 were found embedded in the inside surface of the cladding, as illustrated in Fig. 10, but there was no evidence of reaction between the UO_2 and the stainless steel.

The cladding of capsule E-11 contained an area of considerable intergranular porosity; this region is compared to a more typical region, which had much less porosity, in Fig. 11. We saw no significant changes in the microstructure of the UO_2 .

The apparent roughness of the cladding of capsule E-12R was verified by metallographic examination. This surface roughness and general intergranular porosity are shown in Fig. 12. In addition, this figure indicates that the UO_2 experienced some grain growth in the interior regions of the pellets; the average grain size at the inside surface was about twice that at the outside surface.

EXAMINATION OF TUBES WHICH CONTAINED NaK

The type 304 stainless steel NaK tubes from ETR-irradiated capsules E-5R, E-11, and E-12R were examined to determine the presence and extent of nitriding. [Nitrogen had been used as a heat transfer gas, and the NaK tubes had operated at a temperature conducive to nitriding ($\sim 1000^\circ\text{F}$), so some nitriding was expected.] Severe nitriding of the NaK tube from

R-25178

R-25176

0.035 INCHES
100X

0.035 INCHES
100X

Fig. 8. Section Through the Fracture Surfaces of Capsule E-3. Note the ferritic layer and the cup-cone shape of the fracture surfaces. 100X, Etched.

(a)

0.035 INCHES
100X

(b)

0.035 INCHES
100X

Fig. 9. Comparison of UO_2 from Central (a) Region and Peripheral (b) Region of the Solid Pellet. Note the elongated grains in the central region. 100X, Etched.

Fig. 10. Section of Capsule E-3 Fuel and Cladding Where Small Pieces of UO₂ had been Embedded in Stainless Steel.

Fig. 11. Two Regions from Cladding of Capsule E-11, Showing (a) an Unusual Amount of Intergranular Porosity, and (b) a More Typical Region, With Much Less Porosity. 100X, Etched.

Fig. 12. Two Regions from Capsule E-12R. Note the cladding surface roughness, the general intergranular porosity in the cladding, and the grain size increase across the fuel pellet radius. 100X, Etched.

capsule 02A-6, which was irradiated under similar conditions, was found and reported previously.

As in NaK tubes from previous prototype experiments, the exterior surfaces of these tubes were darkened in the area adjacent to the fuel capsules. This appearance is shown in Fig. 13. Diameter measurements showed consistent increases, up to a maximum of 0.042 in. at one location. The increases in the average diameters are shown in Table 4.

Table 4. Diameter Data for NaK Tubes from EGCR
Prototype Capsules

Measurement Position (in. from bottom end)	Average Diameter Increases ^a		
	E-5R (in.)	E-11 (in.)	E-12R (in.)
0	0.007	0.002	0.004
1	0.006	0.002	0.023
3	0.014	0.002	0.031
5	0.020	0.008	0.020
7	0.001	0.011	0.003
9	0.001	0.003	0.001

^aSince no specific preirradiation measurements were made, these data assume a fabrication diameter dimension of 0.980 in.

A transverse section was selected for metallographic examination from the maximum diameter region of each of the three NaK containers. Since the containers were of a constant diameter prior to irradiation, we presumed that the maximum diameter region would exhibit the maximum extent of nitriding, or of whatever else had caused the diameter changes, which are shown in Table 4.

The microstructure of the NaK container from capsule E-5R is shown in Fig. 14; this region was 5 in. from the bottom end of the container. The nitride case was about 0.008-in. thick, and the maximum nitride penetration was about 0.030-in. A small amount of sigma phase was present

R-22860

Fig. 13. Appearance of a Typical NaK Tube. Note the area of discoloration which shows the location of the fuel capsule inside.

Fig. 14. Transverse Section from E-5R NaK Container, Showing Nitriding in the Outside Surface, (a) As Polished, and (b) Etched. 100X.

throughout the tube wall thickness.

Examination of the section taken 7 in. from the bottom end of the capsule E-11 NaK container disclosed no evidence of nitride or of sigma phase (Fig. 15). The microstructure appeared to be entirely normal with no unusual features.

The section from capsule E-12R was taken 3 in. from the bottom end of the NaK container. As may be seen in Fig. 16, the nitride case was about 0.018-in. thick, and some nitride had penetrated to depths of 0.030 in. In addition, a gross amount of sigma phase was observed throughout.

DISCUSSION OF RESULTS

With two exceptions, the visual appearance of these nine capsules was normal. Based on the extensive cladding distortion observed in capsule E-12R, we must conclude that this capsule had experienced unusually strenuous operating conditions;^{9,10} i.e., cladding temperature and thermal cycling in excess of the design conditions. The survival of this capsule without cladding fracture is evidence of a reasonable safety factor in the design, at least for this particular set of conditions. The drastic damage suffered by capsule E-3 is similar to and consistent with the failures observed in other EGCR prototype capsules which were operated appreciably above design conditions.²

The only unusual feature of the gamma scans was the considerable axial gradient of capsule E-12R. Assuming that the radioactivity gradient was caused by a neutron flux gradient, it seems likely that the resultant heat flux gradient contributed to the cladding distortion discussed above, as well as the general swelling (both radial and axial) which was detected by dimensional measurements. The small dimensional changes ($\sim 1\%$) seen in most of the capsules was not considered to be detrimental; experience had indicated that diameter increases of at least 3% are generally required to cause cladding failures. Capsule E-3, which did rupture, was swollen to this extent or more over about half its length, and the swelling near the fracture was about 8%. The small diameter decreases of capsules E-4R, E-10, and E-11 indicate that the cladding collapsed against the fuel pellets, as expected, because of the 300 psia external pressure during irradiation.

Outside Surface

Inside Surface

Fig. 15. Transverse Section from E-11 NaK Container, Which was Not Nitrided. 100X, Etched.

Outside Surface

Inside Surface

Fig. 16. Transverse Section from E-12R Container, Showing Extreme Nitriding in the Outside Surface, (a) As Polished, and (b) Etched. 100X.

Examination of the cladding fracture region in capsule E-3 showed that the failure resulted from fuel swelling, rather than from embrittlement of the cladding. The shape of the fractured surfaces indicated that no significant loss of ductility beyond that resulting from neutron irradiation^{11,12} had occurred, and no detrimental precipitates were seen in the microstructure. The ferritic layer on the outside surface of the cladding was caused by leaching of the chromium from the stainless steel by the NaK. The existence of this ferritic layer is an indication that the cladding experienced temperatures higher than the design temperature of 1600°F, especially in the region of the fracture. Although small amounts of ferrite have been seen in the cladding of a few other EGCR prototype capsules, this was the most extensive case. In addition, the presence of the ferrite in the fractured surfaces showed that the irradiation continued for some appreciable time after the fracture occurred. The columnar-grained region in the interior of the UO₂ pellet was firm evidence that the fuel had operated in excess of design temperature¹³ (~ 3000°F vs 2400°F).

The only unusual feature seen in the microstructure of capsule E-11 was the intergranular porosity in the cladding, the extent of which varied locally. Since there was no evidence of appreciable cladding deformation, which was found to have caused cladding porosity in previous capsules,¹² no explanation for this porosity was apparent. The extensive cladding deformation seen in capsule E-12R was presumed to be the primary cause of the observed surface roughness and intergranular porosity. The increase in grain size across the fuel pellet radius in capsule E-12R appeared to be the result of temperature-induced grain growth, indicating that the central region of the pellet reached a temperature of ~ 2800°F sometime during the irradiation.

The condition of the facility tubes, which contained NaK, was not surprising in view of the operating conditions. In two of the three cases studied, nitrogen in the nitrogen-argon heat transfer gas had reacted with the stainless steel, effecting nitride precipitation (presumably CrN and Cr₂N were the primary reaction products)¹⁴ to depths of 0.030 in. Although nitrogen had been present in the gas for a significant time in all three capsules, the NaK tube in capsule E-11 was not nitrided.

Apparently, at the combination of lower nitrogen fraction ($\sim 25\%$ vs $\sim 75\%$) and lower temperature for capsule E-11, as compared to capsules E-5R and E-12R, the nitriding reaction did not proceed. Since neither the detailed history of composition of the gas mixtures nor accurate temperature data are available (no temperature measurements of the NaK tubes were made), conclusions about the conditions of nitride formation in these capsules are limited. The two capsules with heavily nitrided NaK tubes operated at average temperatures of about 1450°F (Table 2), approximately 200°F higher than the assembly which was not nitrided. Heat transfer calculations indicate a temperature drop between the capsule wall ($\sim 1450^{\circ}\text{F}$) and the outside surface of the NaK tube of about 100°F . Thus the average temperature of the nitrided surfaces must have been about 1350°F compared to about 1150°F for the surface which was not nitrided.

CONCLUSIONS

Eight of the nine irradiation capsules performed satisfactorily. Although capsule E-12R experienced considerable cladding distortion, and several other capsules were deformed to lesser extents, only capsule E-3 suffered cladding fractures. The cause of failure of this capsule appeared to be identical to the cause of failure of previous EGCR prototype capsules; overheating of the UO_2 fuel pellets caused expansion sufficient to exceed the elastic limit of the stainless steel cladding, resulting in fractures in the region of maximum stress.

Two of the three NaK tubes examined were heavily nitrided, as expected. The third tube, which operated at somewhat lower temperature and nitrogen partial pressure, was not nitrided.

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*Mr. Manly is now associated with the Stellite Division, Union Carbide Corporation.

irradiation examination was directed by H. E. Robertson of the Reactor Chemistry Division. The examination and evaluation was conducted and this report was written under the supervision of O. Sisman.

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