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THE FABRICATION AND IRRADIATION TESTING OF DISPERSION-
TYPE BeO-UO₂ CERAMIC FUEL MATERIALS

by

D. E. Johnson and F. H. Lofftus

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This paper was presented at the Pacific Coast Northwest Regional American Ceramic Society Meeting, Nuclear Ceramics Session, Seattle, Washington, October 17-19, 1962.

ABSTRACT

Considerations affecting the design of dispersion-type ceramic fuel materials are discussed. The results of measurements made to study the effect of fuel loading on the properties of the fuel bodies and the results of irradiation tests of fuel pellets composed of 20 vol-% UO_2 dispersed in BeO are presented. Experience at General Atomic in the fabrication of dispersion-type BeO-UO_2 ceramic fuel materials is described. From the results obtained, it is believed that BeO-UO_2 fuel materials of the type studied will exhibit satisfactory performance in the Experimental Beryllium Oxide Reactor.

I. INTRODUCTION

The use of beryllia as a fuel diluent in dispersion-type, high-temperature, power-reactor fuel materials is potentially advantageous as compared with the use of undiluted UO_2 . For example, if a major fraction of the fuel material is beryllia, a substantially lower average fuel temperature would be expected during reactor operation because of the outstanding thermal conductivity of the beryllia as compared with other oxide ceramic materials. The lower average fuel temperature should permit operation of the reactor at higher fuel-surface temperatures. The fractional release of fission products from the fuel bodies should be minimized because of the lower fuel temperatures, since the diffusion process is quite sensitive to temperature. Also, the dense beryllia matrix should present a barrier that will retard or prevent the escape of fission products from the enclosed fuel particles to the surface of the fuel bodies.

One of the fuel materials that has been under consideration for use in the Experimental Beryllium Oxide Reactor (EBOR) Program¹ is UO_2 diluted with 70 to 80 vol-% of BeO . The results obtained in the study of this fuel system are summarized in this paper.

II. SELECTION OF FUEL LOADING AND FUEL PARTICLE SIZE

Many of the factors affecting the choice of fuel-particle size and fuel loading have been discussed by Weber,² Simnad and Wallace,³ and Hickman.⁴ In addition, the escape of the fission-product xenon from fused and from sintered particles of UO_2 has been reported by Booth and Rymer.⁵ Some of the results of these investigations are reviewed in Figs. 1 and 2.

Figure 1 illustrates some of the more important factors that affect the selection of UO_2 particle size for dispersion-type fuels. Experimental

Fig. 1. Xenon diffusion, fission fragment recoil escape, and radial temperature drop for UO_2 fuel particles

Fig. 2. Volume fraction of undamaged BeO matrix as a function of the volume fraction of the dispersed phase (Ref. 4)

data of Booth and Rymer for the escape of xenon by diffusion from particles of fused UO_2 are presented, together with the theoretical curves. From these experimental data, it was found that for isothermal annealing the quantity of xenon released in a given time was inversely proportional to the UO_2 particle radius. Thus, in order to minimize the rate of escape by diffusion of fission products from the UO_2 particles, it is desirable to use large fuel particles. However, since diffusion rates are strongly temperature-sensitive, the temperature drop across the radius of a UO_2 particle should be considered in arriving at a maximum particle-size limitation. For gas-cooled reactors, such as the Experimental Beryllium Oxide Reactor, where the specific heating rates in the fuels are relatively low, this factor may be of minor importance. For example, at a heating rate of 2000 w/cm^3 in the UO_2 a temperature drop of 100°C would be expected across the radius of a $1400\text{-}\mu$ -diameter particle.

The escape of fission fragments by recoil from the fuel particles is also strongly influenced by the size of the particles. From Fig. 1 it may be observed that a spherical UO_2 particle diameter of approximately 150μ is required if the release of fission fragments from the UO_2 by recoil is to be limited to 10% of the number produced. Below this UO_2 particle diameter the fractional release by recoil increases rapidly with decreasing particle diameter. Related to the recoil escape of fission fragments from the fuel particles is their effect on the matrix material. Figure 2 illustrates the relationship between the volume fraction of a BeO matrix that remains undamaged by the recoils and the volume fraction of the dispersed UO_2 phase for several spherical UO_2 particle sizes. Damage to the matrix material is clearly reduced by utilizing the larger particles of UO_2 .

In an effort to identify some of the limitations associated with the volume fraction of UO_2 that can be dispersed in a practical ceramic matrix, electrical resistance measurements were made at General Atomic on compacts containing a range of UO_2 loadings. Both fine (1 to 10μ average particle diameter) and coarse ($\sim 800 \mu$ average particle diameter) dispersions

were studied. The specimens were made by methods similar to those described in Section III. The results of the measurements are presented in Fig. 3. The shape of the curves is explained on the basis of the relative electrical resistivities of the alumina and UO_2 phases. Since the electrical resistivity of alumina is approximately 10^8 times that of UO_2 , a change in electrical resistance as a function of composition is an indication of the relative fraction of the UO_2 particles that are isolated from each other by the matrix material. It has been concluded from these measurements that for conventional fabrication methods the volume fraction of UO_2 should be limited to approximately 30 vol-% and that a dispersion of coarse particles is superior to a dispersion of fine particles for UO_2 volume fractions greater than approximately 20%. For UO_2 loadings greater than 25 or 30 vol-%, a significant fraction of the UO_2 particles may be in contact with each other. Since UO_2 is mutually insoluble with either alumina or beryllia, it is expected that the interface between the fuel and matrix phases would be a path for easy escape of fission products from the fuel bodies if extensive UO_2 particle-to-particle contact exists.

III. FABRICATION OF BeO-UO_2 FUEL COMPACTS

In general, the problems encountered in the fabrication of dispersion-type fuel bodies are similar to those for other dispersion-type fuel systems, such as cermets. First, in order to limit the fission-fragment recoil damage to the region of the matrix immediately surrounding the fuel particles, thereby preserving the maximum volume fraction of the matrix material in a relatively unaffected condition, it is necessary to prevent "dusting" of the fuel during the process of blending the fuel particles into the matrix material. Second, the fuel particles must shrink at a rate and by an amount that is equal to or greater than those for the matrix material in order to prevent excessive stresses or cracking in the sintered matrix. Third, the shape of the fuel particles ideally should be spherical in order to present the

Fig. 3. Effect of UO_2 particle size and UO_2 volume fraction on the electrical resistance of Al_2O_3 - UO_2 specimens.

smallest surface-to-volume ratio, thereby minimizing fission-product escape from the fuel particles by recoil and diffusion, as well as minimizing the volume fraction of the matrix that may be damaged by recoils. Lastly, the fuel particles should be distributed uniformly throughout the matrix in order to assure uniform macroscopic heat generation in the fuel bodies.

The general features of a typical flow sheet for the fabrication of dispersion-type BeO- UO_2 fuel pellets are illustrated in Fig. 4. A photomicrograph of typical pellets composed of BeO plus 25 vol-% UO_2 particles that were fabricated at General Atomic is presented in Fig. 5. The UO_2 particle size was approximately 300 μ . It may be observed that a random uniform dispersion of the UO_2 particles, which are angular in shape rather than the ideal spherical geometry, was obtained. These results represent a compromise that was made to simplify the fabrication procedure.

"Dusting" of the fuel particles during the blending operation has been minimized by careful processing, with the result that powder and loosely attached small UO_2 particles are removed from the UO_2 particles before the blending operation is performed. The powder and small particles are collected and reprocessed. In general, cracking of the BeO matrix during sintering due to incorrect relative shrinkage of the BeO and UO_2 phases has not been a serious problem, even though the shrinkage on a macro scale is usually of the order of 18% to 22%. Problems that do occur because of the relative shrinkage of the two powders can usually be overcome by appropriate calcining of the starting powders. Sintered densities in excess of 95% of theoretical density have been readily obtained.

IV. IRRADIATION TEST RESULTS

Four capsules containing fuel pellets with nominal compositions of 20 vol-% UO_2 have been irradiated and examined as a part of the Experimental Beryllium Oxide Reactor Program. A detailed description of the methods and materials used has been reported.⁶ The first capsule was

Fig. 4. Flow sheet for fabrication of BeO- UO_2 fuel pellets

Neg. 1675

Fig. 5. Photomacrograph of typical
BeO-25 vol-% UO_2 fuel pellets

irradiated in the Battelle Research Reactor for eight weeks to a fission burnup of approximately 1.4% of the uranium atoms present.³ The remaining three capsules were irradiated in the Materials Testing Reactor for periods of 225-1/2, 297, and 429 full-power reactor days to fission burnups of approximately 4.4%, 5.5%, and 11%, respectively, of the uranium atoms present. The temperature of the fuel pellets for these experiments was estimated to be approximately 1750° to 1875°F, assuming that there was only helium in the 5- to 6-mil diametral gas gap between the pellets and their stainless steel claddings. A similar calculation, based on the assumption that nitrogen occupied the gas gap between the pellets and their claddings, yielded pellet temperatures of approximately 2200° to 2400°F. The true average pellet temperatures were probably between these extremes because of the effect on the conductivity of the helium due to the release of krypton and xenon from the fuel pellets during irradiation.

The results of dimensional and density measurements of the BeO-UO₂ pellets before and after irradiation are presented in Table 1. From these data it has been concluded that the dimensional changes that resulted from irradiation are within tolerable limits for high-temperature, gas-cooled reactor applications. The maximum dimensional change found was an increase in the length of 3%. Most of the dimensional changes were in the range of 1% to 2% or less. Some pellets exhibited decreases in their dimensions; the reason for this behavior is unknown.

Metallographic examination of selected specimens did not reveal any major changes in the microstructure that might have resulted from irradiation. Typical examples of the metallographic study results are presented in Figs. 6 through 15. Qualitative evidence that some structural change in the pellets resulted from the irradiation was obtained during the disassembly of the capsules in the hot cell; it was found that the pellets could be abraded with a file, as illustrated in Fig. 8. Attempts to similarly abrade an unirradiated pellet were unsuccessful, as would be expected. It was also found that the metallographic preparation of irradiated pellets was more

Table 1
DIMENSIONS AND DENSITIES OF BeO-UO₂ FUEL PELLETS BEFORE AND AFTER IRRADIATION

Pellet No.	Fraction of UO ₂ (vol-%)	Estimated Burnup		Dimensional Measurements						Measured Density				
				Diameter			Length			Preirradiation		CCl ₄ Displacement (g/cm ³)	Postirrad. Density (g/cm ³) ^a	Change (%) ^b
										Xylene Absorption and Displacement				
		Pre-irrad. (in.)	Post-irrad. (in.)	Change (%)	Pre-irrad. (in.)	Post-irrad. (in.)	Change (%)	(% of T. D.)	(g/cm ³)					
1-1	24.	14,400	0.87	0.2220	0.2220	Nil	-----	-----	----	----	----	----	----	----
1-2				0.2217	0.2217	Nil	-----	-----	----	----	----	----	----	----
1-3				0.2219	0.2218	Nil	-----	-----	----	----	----	----	----	----
1-4				0.2220	0.2219	Nil	-----	-----	----	----	----	----	----	----
15171-50-6	19.1	37,700	2.0	0.2218	0.2223	0.2	0.2494	0.2515	1.0	94.5 ^c	4.38	4.412	----	----
-7				0.2226	0.2225	Nil	0.2499	0.2510	0.5	94.0 ^c	4.36	4.399	----	----
-10				0.222	0.2225	0.2	0.2485	0.2505	1.0	93.8 ^c	4.35	4.388	----	----
-12				0.2223	0.2225	0.1	0.2508	0.2505	-0.1	93.8	4.35	4.385	----	----
15171-50-13	19.1	37,700	2.0	0.222	0.2203	-1.0	0.2503	0.2503	Nil	95.2 ^c	4.42	4.454	4.37	-1.
-14				0.2241	0.2221	-1.0	0.2493	0.2501	0.4	93.1 ^c	4.32	4.358	4.30	-0.5
-17				0.2229	0.222	-0.4	0.2501	0.2505	0.2	93.1 ^c	4.32	4.369	4.33	0.15
-24				0.2227	0.2223	-0.2	0.2510	0.251	Nil	93.1 ^c	4.32	4.351	4.33	0.15
15171-31-8	22.6	44,800	2.6	0.222	0.227	2.2	0.246	0.253	-2.8	89.6 ^d	4.31	4.39	4.50	+2.5
-9				0.225	0.228	1.3	0.250	0.256	2.4	89.1 ^d	4.29	4.36	4.34	-0.46
-12				0.222	0.226	1.8	0.251	0.254	1.2	89.3 ^d	4.30	4.37	4.32	-1.1
-17				0.222	0.226	1.8	0.249	0.252	1.2	89.8 ^d	4.32	4.38	4.24	-3.2
15171-50-2	20.4	48,800	2.7	0.2226	0.2218	-0.36	0.2411	0.2484	3.0	93.8 ^c	4.35	4.38	4.34	-0.91
-8				0.2224	0.2211	-0.58	0.2500	0.2488	-0.48	94.8 ^c	4.40	4.44	4.39	-1.1
-21				0.2223	0.221	-0.58	0.2502	0.2442	-0.4	94.0 ^c	4.36	4.39	4.33	-1.4
-22				0.2221	0.2212	-0.40	0.2479	0.2504	1.0	93.8 ^c	4.35	4.38	4.34	0.91
15171-31-5	22.6	96,300	5.8	0.221	-----	----	0.239	-----	----	90.5 ^d	4.35	4.44	----	----
-13				0.222	-----	----	0.251	-----	----	90.8 ^d	4.37	4.47	----	----
-30				0.222	-----	----	0.251	-----	----	90.0 ^d	4.33	4.40	----	----
-31				0.222	0.2275	2.5	0.249	0.2519	1.2	89.4 ^d	4.30	4.36	4.12	-5.5
15171-50-3	20.4	92,200	5.1	0.2217	0.2234	0.77	0.243	0.2483	2.2	95.1 ^c	4.41	4.44	4.21	-5.4
-5				0.2228	0.2232	0.17	0.2498	0.2520	0.88	95.0 ^c	4.40	4.44	4.20	-5.4
-16				0.2219	-----	----	0.251	-----	----	92.5 ^c	4.29	4.33	----	----
-19				0.2228	0.2265	1.7	0.2512	0.2536	0.96	93.0 ^c	4.31	4.24	4.13	-2.6

^aMeasured by water immersion and absorption technique.

^bChange in density calculated from the CCl₄ displacement measurements

^cBased on calculated theoretical density of 4.64 g/cm³.

^dBased on calculated theoretical density of 4.81 g/cm³.

Neg. RM 13367

| 200 μ |

Fig. 6. Photomicrograph of a typical area of pellet showing angular UO_2 particles dispersed in BeO ; 19.1 vol-% UO_2 ; as polished, unirradiated

Unirradiated

Neg. N 59585

100 μ

Irradiated

Neg. M 0045

100 μ

Fig. 7. Photomicrographs of typical BeO-19.1 vol-% UO₂ fuel pellets showing BeO grain structure before and after irradiation to $\sim 2 \times 10^{20}$ fissions/cm³ of pellet; etched in 25% NH₄F·HF

Neg. S 0147

Neg. S 0145

Fig. 8. Photomacrographs of an irradiated BeO-19.1 vol-% UO_2 fuel pellet after irradiation to $\sim 2 \times 10^{20}$ fissions/cm³ of pellet; note the small flat caused by filing the surface of the pellet

Neg. M 0060

25 μ

Fig. 9. Photomicrograph of a BeO-19.1 vol-% UO₂ fuel pellet after irradiation to $\sim 2 \times 10^{20}$ fissions/cm³ of pellet, showing grain structure of the BeO between two adjacent UO₂ particles; etched with 25% NH₄F · HF

Neg. RM 13364

100 μ

Fig. 10. Photomicrograph of a typical area in a BeO-20.4 vol-% UO_2 pellet before irradiation; etched in a solution of 1 part H_2SO_4 and 9 parts H_2O_2

Unetched

Neg. M 0616

50 μ

Etched

Neg. M 0627

50 μ

Fig. 11. Photomicrographs of one area of a BeO-20.4 vol-% UO_2 pellet before and after etching in a solution of 1 part H_2SO_4 and 9 parts H_2O_2

Unirradiated

Neg. RM 13363

200 μ

Irradiated

Neg. M 0626

200 μ

Fig. 12. Photomicrographs of typical areas in a BeO-20.4 vol-% UO_2 before and after irradiation to $\sim 2.7 \times 10^{20}$ fissions/ cm^3 of pellet; etched in a solution of 1 part H_2SO_4 and 9 parts H_2O_2

Unirradiated

Neg. RM 13361

200 μ

Irradiated

Neg. M 0601

200 μ

Fig. 13. Photomicrographs of typical areas in BeO-20.4 vol-% UO₂ before and after irradiation to $\sim 5.1 \times 10^{20}$ fissions/cm³ of pellet; as polished

Neg. S 0478

Neg. S 0476

Neg. S 0477

Fig. 14. Photomacrographs of a BeO-20.4 vol-% UO₂ fuel pellet after irradiation to $\sim 5.1 \times 10^{20}$ fissions/cm³ of pellet; the unusual appearance of the pellet surfaces is believed to be the result of exposure to NaK during part of the irradiation

Etched Neg. RM 13362
Unirradiated

As polished Neg. M 0589
Irradiated

Etched Neg. M 0593
Irradiated

100 μ

Fig. 15. Photomicrographs of BeO-20.4 vol-% UO_2 before and after irradiation to $\sim 5.1 \times 10^{20}$ fissions/ cm^3 of pellet; etching was performed using a solution of 1 part H_2SO_4 and 9 parts H_2O_2

difficult than for unirradiated pellets because of "pull-out" of BeO grains, as illustrated in Fig. 15. In fact, the lower-density pellets that had been irradiated to approximately 5.8×10^{20} fissions/cm³ could not be satisfactorily polished.

The grain structure of the BeO between two particles of UO₂ after irradiation to $\sim 2 \times 10^{20}$ fission/cm³ of pellet is illustrated in Fig. 9. This grain structure is similar to that observed in the unirradiated material (Fig. 7). Also, the results of X-ray diffraction measurements⁶ indicated that the BeO was still crystalline after the irradiation. These results are in direct contrast with those reported by Yeniscavich and Bleiberg⁷ for BeO-UO₂ and by Berman, Bleiberg, and Yeniscavich⁸ for Al₂O₃-UO₂. It is suggested that the observed difference in behavior may be the result of substantially higher material temperatures in the present irradiations. A BeO grain structure similar to that illustrated in Fig. 9 was found in the BeO in pellets irradiated to $\sim 2.7 \times 10^{20}$ fissions/cm³ of pellet. Efforts to etch the BeO in the more heavily irradiated material (5.1×10^{20} fissions/cm³) were unsatisfactory because of the porosity resulting from the "pull-out." Similar observations for the more heavily irradiated material have therefore not been made.

Some caution is required in analyzing the dimensional data presented in Table 1 for the most highly irradiated specimens. This is necessary because the stainless steel pellet claddings ruptured during the irradiation, with the result that NaK was permitted to come in contact with the pellets. The appearance of these pellets after irradiation is illustrated in Fig. 14. It may be seen that a loose, friable scale was formed on the pellet surfaces. This scale was removed by brushing before the measurements were made, and it must be assumed that this may have had some effect on the precision of the measurements. However, it is believed that for purposes of evaluating the material for reactor use the results are adequately precise, particularly since fair agreement between dimensional measurement results and density measurement results was obtained.

V. CONCLUSIONS

As a result of these irradiation tests, the following conclusions have been reached:

1. The volume fraction of UO_2 in BeO-UO_2 fuel pellets should not exceed 25% to 30% if optimum performance is to be obtained.
2. The UO_2 particles in BeO-UO_2 fuel bodies should not be less than approximately $125\ \mu$ in diameter.
3. $\text{BeO-20 vol-\% UO}_2$ fuel pellets with the UO_2 present as $125\text{-}\mu$ angular grains is suitable for use as a high-temperature reactor fuel material and can satisfactorily withstand reasonable irradiation exposures.
4. A comparison of the results obtained in this study with some of the results obtained by others indicates that the performance of BeO-UO_2 may be improved as a result of high material temperatures during irradiation. It is not yet possible to define specific temperature limitations, if they do exist.
5. In a qualitative sense, it was found that the resistance of the BeO-UO_2 fuel pellets to abrasion was reduced as a result of irradiation. Reasons for this change were not determined.

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