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A PROGRESS REPORT

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Quarterly Report on the
Space Electric Power R and D Program
for the Period Ending July 31, 1972

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QUARTERLY REPORT ON THE
SPACE ELECTRIC POWER R AND D PROGRAM
For the Period Ending July 31, 1972

SUMMARY

Five ceramic-metal multilayers have been fabricated for inclusion in the Experimental Breeder Reactor (EBR-II) and are being assembled into Isothermal Irradiator Capsule K-5, together with 11 specimens from Gulf General Atomic Co. (GGA). Difficulties in testing the heat pipe for this capsule have been overcome, and tests of the prototype unit show that, with the pipe operating at 745°C, a threefold increase in power transmitted down the pipe increases the operating temperature by ~ 30°C.

Guard-ring measurements of the specific resistance of yttria that has been incorporated into a Nb-1%Zr sheathed trilayer unit by plasma-arc spraying and subsequent autoclaving indicate that at 750°C an electric field of 400 V/cm causes the resistance to decrease with time. After 800 h the specific resistance has dropped to ~ $4 \times 10^4 \Omega \text{ cm}$; the rate of decrease at this time was much less than the initial rate but was still nonnegligible.

Disassembly of EBR-II Irradiation Capsules K-3 and K-4 has been completed and posttest diagnostic measurements are in progress. Initial dimensional increase data show that cylindrical shell specimens of Al_2O_3 with an average grain size of 2 μm and of Al_2O_3 -30v/oNb cermet are equivalent in dimensional stability and that both may be slightly more stable than the 5- μm alumina tested earlier. A sapphire specimen irradiated at ~ 750°C increased dimensionally almost exclusively along the c-axis of the crystal structure. Yttria with an addition of 10% ZrO_2 showed no dimensional increase.

Visual examination of trilayer specimens from Capsules K-3 and K-4 revealed that ungraded Al_2O_3 over Nb-1%Zr bonds separated even at neutron fluences below $2 \times 10^{21} \text{ n/cm}^2$ ($E_n > 0.1 \text{ MeV}$). Ungraded Al_2O_3 over niobium bonds were more resistant to neutron damage, but partial debonding occurred for neutron fluences of $2.7 \times 10^{21} \text{ n/cm}^2$ and greater. No separation was seen for trilayers with graded Al_2O_3 -Nb bonds, but exposures were limited to $2.5 \times 10^{21} \text{ n/cm}^2$. Trilayers fabricated with Y_2O_3 and either niobium or Nb-1%Zr revealed no obvious debonding at neutron fluences up to $4.8 \times 10^{21} \text{ n/cm}^2$, whether graded or not. A trilayer specimen fabricated with an Al_2O_3 -30v/oNb cermet seemed undamaged after having been exposed to a neutron fluence of $3.7 \times 10^{21} \text{ n/cm}^2$ at 750°C.

Metallic inclusions in UO_2 irradiated in contact with CVD tungsten for 10 178 h at 1600°C have been identified by electron-microprobe examination to be tungsten. Both tungsten and metallic fission-product alloy inclusions have been identified in the UO_2 of a tungsten-clad thermionic emitter that operated at open circuit for 76 h near the end of a 1374-h irradiation test.

SHEATHED INSULATOR DEVELOPMENT

Fabrication of Multilayers

The five prototypical multilayer specimens to be tested in EBR-II Irradiation Capsule K-5 have been pressure-bonded and machined to final dimensions. The general configuration of both the trilayer and pentalayer specimens is shown in Fig. 1. The radial scale is exaggerated to illustrate more clearly the relative thicknesses of the ceramic and metal layers. The region of the pentalayer thermionic fuel-rod sheath design where the metal interlayer connects to the collector has been included in two of the three pentalayer test specimens because this is the area in which fast-neutron damage to the ceramic is most likely to occur.

Fabrication details are given in Table I. The two trilayer specimens (LDTL-140 and -141) were designed to test the comparative effect on Y_2O_3 -Nb-1%Zr bond integrity of simple grading layers applied by plasma-spraying 50:50-v/o mixtures of yttria and Nb-1%Zr powders. Alumina-based pentalayer Specimens LDPL-137b and -139 were also made to test the

comparative effectiveness of arc-sprayed cermet grading layers. LDPL-137b was prepared without grading layers, and LDPL-139 had these layers applied only at those interfaces at which the ceramic encloses the metal components, because here the bonds are put into tension when the alumina expands as a result of fast-neutron damage. Pentlayer LDPL-135 represents a different approach to grading: The Nb-1%Zr interlayer is enclosed by an yttria layer followed by a somewhat thinner alumina layer. Because yttria does not expand under irradiation, this joint should experience little tensile stress. Introduction of an alumina layer between the yttria and the outer sheath will increase the electrical resistance of the ceramic layer in this region of relatively high electric fields. (In the pentalayer design the electric field across the inner ceramic layer is relatively low so that the use of yttria in this region should cause no problems.)

Several units, which could be tested in EBR-II Irradiation Capsule K-6, are being machined to final dimensions. The units to be included will be selected after more diagnostic measurements are available from Irradiation Capsules K-3 and K-4 discussed later in this report.

A single trilayer of prototypical size containing arc-plasma sprayed $Y_3Al_5O_{12}$, and several small-diameter trilayers containing either this compound or Y_2O_3 , have been fabricated. The prototypical trilayer will be used for irradiation testing of the $Y_3Al_5O_{12}$ compound, and the small-diameter units will be used for tensile and electrical testing. Interest in $Y_3Al_5O_{12}$ was sparked by the fact that this material appears to be a very stable compound of possibly high strength, with a cubic structure similar to that of yttria.

Two other multilayers of prototypical size were also fabricated. One, a Nb-1%Zr- Al_2O_3 design, was autoclaved with a temperature/pressure schedule known to produce multiple cracks in the ceramic. This unit will be tested under an applied voltage in cesium vapor at 750°C to determine the effectiveness of the pentalayer design in eliminating the potential cesium arcing problem caused by cracks in the ceramic sheaths or in the ceramic coatings of typical thermionic fuel-rod sheath designs. The second unit is an yttria trilayer with thin plasma-

Fig. 1. Configuration of LASL trilayer and pentalayer specimens for EBR-II Irradiation Capsule K-5. (All dimensions in millimeters.)

TABLE I
MULTILAYER SPECIMENS FOR EBR-II ISOTHERMAL
IRRADIATOR CAPSULE K-5

Composition and Thickness, mm ^a								
No. of Specimen	Collector	Insulator				Outer Sheath		
LDPL-135	Nb-1%Zr	Y ₂ O ₃	Nb-1%Zr	Y ₂ O ₃	Al ₂ O ₃	Nb-1%Zr		
	1.702	0.190	0.076	0.152	0.102	0.762		
LDPL-137-b	Nb-1%Zr	Al ₂ O ₃	Nb-1%Zr	Al ₂ O ₃		Nb-1%Zr		
	1.702	0.190	0.127	0.190		0.762		
LDPL-139	Nb-1%Zr	(50 v/o Nb-1%Zr)		Al ₂ O ₃	Nb-1%Zr	(50 v/o Nb-1%Zr)	Al ₂ O ₃	Nb-1%Zr
	1.702	(50 v/o Al ₂ O ₃)		0.127	0.127	(50 v/o Al ₂ O ₃)	0.127	0.762
		0.051				0.051		
LDPL-140	Nb-1%Zr	(50 v/o Nb-1%Zr)		Y ₂ O ₃	(50 v/o Nb-1%Zr)			Nb-1%Zr
	1.702	(50 v/o Y ₂ O ₃)		0.406	(50 v/o Y ₂ O ₃)			0.762
		0.051			0.051			
LDPL-141	Nb-1%Zr	Y ₂ O ₃						Nb-1%Zr
	1.702	0.508						0.762

^aAll ceramic layers and ceramic/metal mixtures were applied by arc-plasma spraying. Metal interlayers were sprayed except in LDPL-139, which was machined from Nb-1%Zr tubing.

sprayed layers of molybdenum and Mo/Y₂O₃ between the Nb-1%Zr sheaths and the yttria layer. This unit will be used to study the effect of a molybdenum barrier on the stability of the resistance of yttria when the latter is subjected to an electric field at 750°C.

Electrical Resistivity Tests

The apparatus for measuring the effect of voltage-at-temperature on the resistance of yttria trilayers (see LA-4978-PR, Fig. 3) has been modified by including a galvanometer to ensure that the guard rings and the test section of the trilayer are at the same potential. The galvanometer is nulled by varying the resistance between the guard rings and the ground. This replaces the variable potential source originally in this location and appears to be a much better method of compensating for the resistance of the ammeter in the test circuit.

Measurements of the resistance-vs-time behavior of Nb-1%Zr/plasma-sprayed Y₂O₃/Nb-1%Zr trilayers subjected to a voltage of 20 Vdc at 750°C are continuing. The time-dependence of the

specific resistance of the yttria layer observed to date is shown in Fig. 2. Note that resistance decreased by over an order of magnitude during the first 200 h of testing and that at 800 h the specific resistance is still declining, although at a much slower rate. The test was interrupted several times during the early stages because of the formation of electrically conducting deposits on the ceramic surfaces. There is a remote possibility that the cleaning and baking procedures used to remove these deposits could have affected subsequent measurements; additional Y₂O₃ specimens are therefore being fabricated. The drop in resistance of the yttria layer may stem from a reaction between Nb-1%Zr and yttria at the interfaces. This possibility will be investigated by using the trilayer specimen with the molybdenum barrier between the Nb-1%Zr sheaths and the ceramic, described above.

Fig. 2. Effect of applied electric field on specific resistance of yttria at 750°C.

NEUTRON INDUCED DAMAGE TO CERAMICS AND CERAMIC-METAL COMPONENTS

Disassembly of Isothermal Irradiation Capsules K-3 and K-4

Irradiation Capsules K-3 and K-4 were received from the EBR-II on May 25, 1972, after having been irradiated in Grid Position 7C1 for 6331 Mwd. These capsules contained 85 cylindrical-shell ceramic and ceramic-metal trilayer specimens and two Nb-V braze specimens. Several cylinders of niobium, Nb-1%Zr, and TZM alloy were also included. The specimens were mounted in four separate test units: Two were isothermal irradiator assemblies designed to run at 750°C, one was an isothermal irradiator designed for 600°C, and one was designed to maintain the specimens at the coolant temperature of the EBR-II (~ 450°C). The specimens were subjected to neutron fluences ranging from 1.5 to 6.0×10^{21} n/cm² ($E_n > 0.1$ MeV).

Capsule K-4 had been damaged before shipment to IASL: A dent at about the midpoint of the capsule resulted in a 16-mm displacement of one end. X-rays did not reveal any cracks in the specimens due to this damage.

Both capsules, K-3 and K-4, were disassembled and the irradiated specimens have been removed for postirradiation analysis. Each specimen was photographed and visually examined. Among the 87 test specimens, the only severely damaged sample was an yttria insulator that was broken during disassembly by a puncturing tool. All insulator shells had darkened during irradiation but apparently none had cracked.

Measurements of dimensional changes of the ceramic and trilayer specimens have not been completed, but some data exist for the cylindrical-shell ceramic test pieces of greatest interest. These include: Sapphire specimens furnished by Thermo Electron Corp. (TECO) in which the c-axis of the crystals was oriented along the cylindrical axis of the specimens; a specimen of TECO Al₂O₃-30v/o Nb cermet; fine-grained (~ 2 μm) alumina produced by Avco Corp; fine-grained alumina produced by Coors Porcelain Co.; and yttralox, a high-density yttria stabilized with ~ 10% ZrO₂, produced by General Electric Corp. The data are listed in Table II, and volume increase as a function of neutron fluence is plotted in Fig. 3.

The volume increase of the two sapphire specimens is somewhat less than that observed earlier for AD-999 alumina specimens, which had an average grain size of ~ 5 μm. However, it can be seen in Table II that the dimensional increase occurs primarily along the length of the specimen, (i.e., along the c-axis of the Al₂O₃ crystal). At 750°C the dimensional change is almost entirely along this axis. The increase in the ratio of $\Delta c/c \div \Delta a/a$ with increasing irradiation temperature has been noted earlier for solid cylindrical specimens by other investigators.^{1,2}

The Al₂O₃-30v/oNb cermet and the Avco Al₂O₃ specimens showed comparable dimensional increases, with the data largely lying within the dispersion observed earlier for AD-999 specimens. Two AD-999X data points indicate an unexpected behavior i.e., essentially zero length increase. The third specimen behaved as expected -- no essential difference between diametral and length increase. This material has somewhat smaller (~ 4 μm) and more uniform grains than the AD-999 material tested earlier. The data are preliminary and are expected to be better understood when additional tests, including metallography, are completed.

The yttralox specimens showed essentially no dimensional change although the 0.3% shrinkage observed for the specimen irradiated at 750°C may be significant. Earlier investigations by Keilholtz² had found an increase of ~ 0.2% per 10^{21} n/cm² ($E_n > 0.1$ MeV) for solid yttralox cylinders irradiated at 400°C and slightly less than half this amount at 800°C. The reason for the discrepancy is not understood at this time.

TABLE II
INITIAL DIMENSIONAL CHANGE DATA FOR CERAMIC SPECIMENS
IRRADIATED IN CAPSULES K-3 AND K-4

Specimen	Fluence, 10^{21} n/cm ² ($E_n > 0.1$ MeV)	Nominal Irradiation Temp., °C	Diameter Increase, %	Length Increase, %	Volume Increase, %
TECO Sapphire	6.0	450	0.43	1.03	1.90
	5.4	750	0.02	1.70	1.74
TECO Al ₂ O ₃ -Nb Cermets	4.0	750	0.47	0.68	1.62
Avco Al ₂ O ₃	2.8	450	0.34	0.51	1.18
	2.5	750	0.29	0.27	0.86
	4.2	750	0.65	0.68	1.98
AD-999X Al ₂ O ₃	2.6	750	0.37	-0.21	0.54
	4.6	750	0.68	0.95	2.31
	6.4	750	0.83	0	1.66
Ytttralox	2.4	450	-0.05	0.20	0.10
	4.2	750	-0.12	-0.03	-0.27

Fig. 3. Volume expansion versus neutron fluence for selected ceramic specimens from Irradiation Capsules K-3 and K-4.

Twenty-two trilayer specimens irradiated in Capsules K-3 and K-4 had been produced at LASL. These units were constructed by inserting a ground ceramic tube with a 0.5-mm-thick wall between either a niobium or a Nb-1%Zr collector (inner) tube and a 0.2 to 0.25-mm-thick Nb-1%Zr outer sheath, and pressure-bonding the assembly for three hours at 10 000 psi and 1530°C. Nominal outer and inner diameters of these pieces were 15.7 and 12.7 mm, respectively. After being cut to length, the

specimens had the configuration shown in Fig. 4. The unit shown in this figure was irradiated at a nominal temperature of 450°C to a neutron fluence of 2.3×10^{21} n/cm² ($E_n > 0.1$ MeV) and was one of the few alumina-containing specimens that were still intact. Of the eleven alumina trilayers with Nb-1%Zr collectors, eight had clearly debonded at the collector-ceramic (inner) interface, so that the collector could be easily slipped out of the assembly (Fig. 5). Exposures for these eight samples ranged from 1.5 to 5.3×10^{21} n/cm². The three specimens which did not slip easily apart showed definite indications of at least partial debonding. The only suggestion of a temperature effect seemed to exist for the three units constructed with Avco fine-grain (2-3 μm) alumina: One showed complete separation after a fluence of 2.1×10^{21} n/cm² at 600°C, whereas the other two showed only partial debonding for fluences of 2.9 and 3.3×10^{21} at 750°C.

Of the eight LASL trilayer units with alumina ceramic and niobium collectors, four showed partial debonding after exposure to fluences in the range of 2.7 to 4.4×10^{21} n/cm². The other four appeared intact, as in Fig. 4, after having experienced neutron fluences ranging from 1.6 to 2.3×10^{21} n/cm². No indication of a temperature effect was noted.

Fig. 4. Configuration of LASL trilayer specimens in Irradiation Capsules K-3 and K-4. This specimen was irradiated at $\sim 450^\circ\text{C}$ to a neutron fluence of $2.3 \times 10^{21} \text{ n/cm}^2$ ($E_n > 0.1 \text{ MeV}$). (4X)

The irradiation-induced swelling of alumina places the collector-ceramic interface in tension; debonding is therefore expected to occur at some fluence, particularly because of the light restraining action of the thin-walled outer sheath of these specimens. These preliminary results show that debonding can occur at fluences as low as $1.5 \times 10^{21} \text{ n/cm}^2$ for trilayers constructed from fine-grained alumina with Nb-1%Zr collectors. For alumina trilayers with niobium collectors, debonding apparently occurs at fluences between 2.3 and $2.7 \times 10^{21} \text{ n/cm}^2$. More stress-relieving of the collector-ceramic bond was expected to occur with niobium collectors because the creep rate of niobium for a given stress is much higher than that of Nb-1%Zr in the temperature range of these irradiation tests. However, if the difference in bond behavior were the result of creep-rate differences, one would expect to see

Fig. 5. LASL trilayer specimen with Nb-1%Zr collector, ceramic, and Nb-1%Zr outer sheath after irradiation at 750°C to a neutron fluence of $3.0 \times 10^{21} \text{ n/cm}^2$. Separation occurred at collector-ceramic interface. (4X)

some evidence of a temperature effect (because the irradiation-induced swelling rate of alumina is quite insensitive to irradiation temperature). The only evidence of such an effect was cited above for the Avco alumina with Nb-1%Zr collectors. It is largely controverted by the lack of a temperature effect on trilayers having niobium collectors.

These results indicate that in trilayers, in which the ceramic layer is not heavily constrained by a thick outer metal sheath, the partially chemical and partially mechanical bond of Nb-1%Zr to alumina is not sufficiently strong to endure fluences of 4.5 to 5.0 n/cm^2 to which thermionic fuel rods will be exposed in reactors with a fast neutron spectrum. The mechanical bond of niobium to alumina, although somewhat better (for reasons that are not well understood) can only withstand half the required fluence.

Thus other measures, e.g., applying grading layers at the collector-ceramic interface and more strongly restraining the ceramic by the outer sheath, are necessary at this interface. However, none of the samples revealed any debonding of the outer ceramic-metal interface and strengthening of this bond seems superfluous, i.e., these preliminary results indicate that graded layers are not needed in this region.

Three yttria trilayers were included among the specimens and were subjected to fluences ranging from 1.9 to 4.8×10^{21} n/cm². None showed any debonding, but much more extensive examination is required, as indeed it is for all specimens that are not obviously debonded, before definite conclusions can be drawn.

Eleven trilayer specimens fabricated by GGA were also included in the test capsules. Typically these units had the appearance shown in Fig. 6, differing from LASL specimens in the thickness ratio of outer sheath to ceramic, which was 3:1 rather than 1:2 (LASL). All these units had niobium collectors and outer sheaths. None showed any indication of debonding. The test units included three alumina specimens with successive Nb-25v/o Al₂O₃ | Nb-50v/o Al₂O₃ | Nb-75v/o Al₂O₃ grading layers at each ceramic-to-metal interface and three yttria specimens with a similar Nb-Y₂O₃ grading-layer arrangement. The former were exposed to neutron fluences ranging from 1.7 to 2.5×10^{21} n/cm² and the latter to fluences ranging from 2.5 to 4.0×10^{21} n/cm². Single units containing different types of pure alumina and of alumina with additions of 1% Y₂O₃ and 3% tungsten were irradiated to fluences of 2.1 to 2.2×10^{21} n/cm². All alumina specimens were exposed to the same range of fluences in which the LASL alumina trilayer units with niobium collectors apparently had performed satisfactorily. It is therefore difficult to ascertain from the preliminary examinations how effective grading, restrain, and additives are in preventing the debonding seen in LASL specimens at higher fluences.

The most impressive result observed for an alumina-bonded trilayer was displayed by the single specimen of TECO Al₂O₃-30v/o Nb cermet bonded to a niobium collector and niobium outer sheath (Fig. 7). This specimen was irradiated at a nominal temperature of 750°C to a neutron fluence of 3.7×10^{21} n/cm² and appears to be in very good condition.

The temperatures quoted in the discussion above are design temperatures. To verify that these temperatures were maintained throughout the test, pressure measurements and gas samples were taken from each of the vacuum spaces, the Dewars (which enclosed the specimens), and the heat pipes. The gas pressures were not as expected, but interpretation of these measurements must await the results of gas-sample analyses and of checks on the operation of the gas-collection system.

The fast-neutron fluences to which the insulators in Capsules K-3 and K-4 had been exposed were determined by monitoring the induced activity from the ⁵⁴Fe(n,p)⁵⁴Mn reaction. Stainless-steel samples were taken from both the heat-pipe walls and the outer containment tube. Results are plotted in Fig. 8. Data are not yet available for the upper part of the K-4 capsule because the heat pipe in this section is undergoing other tests. The effective cross section for the ⁵⁴Fe(n,p)⁵⁴Mn reaction used to calculate the fast-neutron fluences was assumed to have a constant value of 87 mb. The variation of this effective cross section for different positions in the reactor and for the reflector change from uranium to stainless steel is being evaluated.

Estimated fluences for $E_n > 0.1$ MeV are plotted in Fig. 9. The flux ratios ($\phi > 0.1$ MeV/ $\phi > 1$ MeV) are those that had been used for K-2 fluences and are based on published neutron spectra for the EBR-II.³ The test specimens thus received the test design fluences of 1.5 to 6.0×10^{21} n/cm², ($E_n > 0.1$ MeV).

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Fig. 6. Configuration of GGA trilayer specimens irradiated in EBR-II Capsules K-3 and K-4. (4X)

Fig. 7. TECO Al_2O_3 -30v/o Nb cermet trilayer specimen after irradiation at 750°C to a neutron fluence of $3.7 \times 10^{21} \text{ n/cm}^2$ ($E_n > 0.1 \text{ MeV}$). (4X)

Fig. 8. Fluence of neutrons ($E_n > 1.0 \text{ MeV}$) versus distance from EBR-II core midplane as determined from $^{54}\text{Fe} (n,p) ^{54}\text{Mn}$ reaction for Irradiation Capsules K-3 and K-4.

Fig. 9. Fluence of neutrons ($E_n > 0.1 \text{ MeV}$) versus distance from EBR-II core midplane for Irradiation Capsules K-3 and K-4.

ISOTHERMAL IRRADIATION CAPSULES

Assembly of EBR-II Irradiation Capsules K-5 and K-6

Experiments with a relatively high-temperature, oil-cooled system for testing the heat pipes for Capsules K-5 and K-6 were described in the last quarterly report (LA-4978-PR). This system provided operating conditions which approximated those encountered in the EBR-II much more closely than previous tests; however, the heat pipes could not be operated with the temperature distributions they will experience during irradiation, and some extrapolation was required to ascertain that the correct amounts of buffer gas and sodium had been included. The oil-cooled system did not provide heat-dump temperatures or heat-removal rates that were as high as anticipated (and required). The limitations on the system were due to an unexpectedly low heat-transfer coefficient between the cooling jacket around the heat pipe and the flowing oil, which resulted in film-boiling and pump cavitation. Because of these difficulties, a different approach to testing these heat pipes has been taken. The system now consists of an rf power supply for heating the evaporator section of the heat pipe; of a copper block, which is clamped tightly to the heat-dump section of the heat pipe (a silver foil is inserted between the copper block and the heat pipe to ensure good thermal contact); and of a water-cooled calorimeter separated from the copper block by a controllable gas gap. Due to the presence of the buffer gas in the heat-dump section of the heat pipe, heat transfer is restricted to a small fraction of the available heat-dump area; however, because of the large heat-transfer coefficient between the heat pipe and sodium coolant of the EBR-II, the surface of the heat-dump region of the heat pipe during reactor operation will be nearly isothermal (approximately at the sodium coolant temperature of $\sim 450^\circ\text{C}$). The copper block, which is clamped around the heat pipe during testing, provides essentially this condition. Both the pressure and the composition (argon-helium mixture) of the gas in the gas gap are controllable, enabling adjustment of the temperature of the surface of the heat-dump section of the heat pipe to that of the reactor coolant for a wide range of heat-removal rates through the calorimeter.

The prototype heat pipe has been tested in this system; both the system and the heat pipe performed very satisfactorily. Figure 10 shows the temperature of the evaporator section of the heat pipe (i.e., the irradiation specimen temperature) as a function of power removed from the heat-dump section of the heat pipe at a constant heat-dump temperature equal, approximately, to the EBR-II coolant temperature. A threefold change in power transmitted through the heat dump resulted in a change of $\sim 30^\circ\text{C}$ in evaporator temperature. The heat pipes for Capsules K-5 and K-6 will be tested in a similar manner.

Approval in principle has been obtained from the Division of Reactor Development and Technology for Capsules K-5 and K-6. Gamma-heating data for the EBR-II with stainless-steel reflector were recently provided by the Argonne National Laboratory, permitting completion of the design of Capsule K-5; the capsule is being fabricated. The nominal specimen irradiation temperature in Capsule K-5 will be 730°C .

The test specimens for Capsule K-5, which were fabricated by GGA and by LASL, were assigned positions in the capsule at a joint meeting between GGA and LASL personnel. Locations of the specimens, relative to the axial midplane of the reactor, together with the relative neutron fluence to which they will be exposed are shown in Fig. 11. The maximum target neutron fluence for this capsule is $6.0 \times 10^{21} \text{ n/cm}^2$ ($E_n > 0.1 \text{ MeV}$). The types of ceramics and ceramic-metal cermets included in the test specimens are listed in Table III. A more complete description of the LASL specimens is contained in the first section of this report.

ORNL Irradiation Capsule KB-2

Some additional data on average grain size and impurity content have been obtained for unirradiated alumina specimens from the same batches that were included in Oak Ridge National Laboratory (ORNL) Irradiation Capsule KB-2. This capsule contained specimens of Coors, WESGO, and opaque Lucalox aluminas and was irradiated for 7572 Mwd in the EBR-II at temperatures in the range of from 815° to 950°C (determined from posttest analyses of SiC temperature monitors). The capsule was disassembled, and posttest diagnostics were performed by LASL.

Fig. 10. Temperature regulation performance of prototype K-5 isothermal irradiator.

Fig. 11. Positions of multilayer specimens in Irradiation Capsule K-5. Ceramic thickness in GGA specimens is 0.381 mm, including the graded metal/ceramic layers. Ceramic thickness in LASL specimens is 0.508 mm, including graded layers and metal interlayer. Specimens are identified in Table III.

TABLE III

MULTILAYER SPECIMENS TO BE INCLUDED IN ISOTHERMAL IRRADIATION CAPSULE K-5

GGA Specimens			LASL Specimens		
Position ^a	Specimen No.	Ceramic	Position ^a	Specimen No.	Ceramic
1	5562-73D	TECO Al ₂ O ₃ -Nb	7	135	Y ₂ O ₃
2	5561-19	Al ₂ O ₃ Graded	8	139	Al ₂ O ₃
3	5561-81B	Al ₂ O ₃ - 15% Mo	9	140	Y ₂ O ₃
4	5561-19	Al ₂ O ₃ Graded	11	141	Y ₂ O ₃
5	5561-29A	Al ₂ O ₃ - 30% W	12	137b	Al ₂ O ₃
6	5561-43B	Al ₂ O ₃ - 10% La ₂ O ₃			
10	4968-125	Y ₂ O ₃ Graded			
13	5561-45B	Al ₂ O ₃ - 10% Y ₂ O ₃			
14	4468-125	Y ₂ O ₃ Graded			
15	5561-29B	Al ₂ O ₃ - 50% W			
16	5562-73D	TECO Al ₂ O ₃ -Nb			

^aSee Fig. 11.

Comparisons between results of measurements of average grain sizes of irradiated specimens from Capsule EB-2 and average grain sizes of unirradiated materials, as specified by ORNL (see LA-4898-PR), indicated that considerable grain growth had occurred during irradiation. Such a result was not very credible and there was concern that either differences in batches or in measurement technique might be involved. Samples of unirradiated materials from batches irradiated in Capsule EB-2 were therefore obtained from ORNL for evaluation. Measurements revealed that average grain sizes were much larger than had been indicated by the initial data. Results of LASL measurements of average grain sizes for both unirradiated and irradiated materials are presented in Table IV.

TABLE IV
RESULTS OF MEASUREMENTS OF AVERAGE
GRAIN SIZES OF ALUMINA SPECIMENS
IRRADIATED IN CAPSULE EB-2

Material	Average Grain Size, μm	
	Unirradiated	Irradiated
GE Opaque Lucalox	17	25 ± 5^a
Coors AD-995	39	48 ± 8
WESGO Al-995	56	54 ± 6

^aStandard deviation from average of measurements of several specimens.

Apparently, no significant grain growth occurred in these materials during irradiation. The differences between results of ORNL and LASL determinations of average grain sizes of unirradiated materials are not readily explained; however, differences in batches of material appear to be the most probable explanation.

FUELED EMITTER DEVELOPMENT

Electron-Microprobe Examination of Emitters SCIP-518 and TFE-621

Electron-microprobe examination of sections of the SCIP-518 emitter and of the center emitter in TFE-621 was completed. Under optical examination, both emitters exhibited metallic inclusions in the UO_2 fuel. The SCIP-518 emitter consisted of alternately stacked wafers of UO_2 and tungsten

enclosed in a CVD-tungsten cladding. The emitter was irradiated for 10 178 h at $\sim 1600^\circ\text{C}$. During this period the powder-metallurgy tungsten disks reacted with the UO_2 , yielding a cermet-type of structure. Little interaction was observed between the CVD-tungsten cladding and the UO_2 , but metallic inclusions were present in the UO_2 adjacent to the cladding surface. Both effects are visible in Fig. 12, which shows a tungsten disk at the top and a small part of the tungsten cladding on the right.

Microprobe examinations revealed that the small metallic particles in the UO_2 adjacent to the tungsten cladding were tungsten. There was no indication of metallic fission products in these inclusions, nor were fission products detected in the region of the tungsten disks.

The TFE-621 emitter was irradiated for 1298 h at a cladding temperature of 1560° to 1585°C , 58 h at $\sim 1930^\circ\text{C}$, and 18 h at $\sim 2100^\circ\text{C}$ with the latter temperature last in the irradiation history. The metallic inclusions observed in the UO_2 in this unit extended from the tungsten cladding to the central void and had the general appearance shown in Fig. 13. The electron-microprobe examination revealed that the inclusions near the central void contained Mo, Tc, Ru, and Rh with small amounts of Pd and U. The inclusions closer to the tungsten cladding contained the first five elements listed above plus tungsten, with increasing tungsten content as the cladding was approached. No evidence of Ce, Nd, Zr, Pr, Nb, Sr, Ba, Cs, Xe, I, or Te was found.

Fission Gas Retention Measurements on TFE-621 Bottom Emitter

Measurements of fission-gas retention in tungsten-clad UO_2 emitters irradiated by General Electric (GE) at Vallecitos, CA, were reported in LA-4898-MS. At that time the fission-gas (^{85}Kr) retention in the bottom emitter of the TFE-621 triple thermionic-diode assembly appeared to be twelve times higher than in the middle and top emitters of this assembly. The measurements have been repeated and yielded a value of 17% for ^{85}Kr retention, which compares favorably with the earlier measurement of 24%. (The corresponding values for the middle and top emitters were 2%).

200 μ

Fig. 12. Junction of powder-metallurgy tungsten-disk and CVD-tungsten cladding of SCIP-518 emitter, showing metallic inclusions in UO_2 adjacent to tungsten cladding. Cladding runs along right-hand side of photograph. (100X)

It seems certain that the high value of ^{85}Kr retention can be ascribed to the existence of a short circuit in the bottom thermionic diode during 245 of the final 274 h at irradiation. This included the 58- and 18-h time intervals near and at the end of the TFE-619 irradiation history when the device was open-circuited and the top and middle emitters operated first at $\sim 1930^\circ C$ and finally at

Fig. 13. Metallic inclusions in UO_2 of TFE-621 middle emitter. (250X)

$\sim 2100^\circ C$. Thus, while the upper two emitters were operating at a temperature high enough to produce almost complete fission-gas venting from the UO_2 , the bottom emitter was operating at a temperature well below the nominal test-temperature range of 1560° to $1585^\circ C$ (probably below $1500^\circ C$), and the average ^{85}Kr retention value of 21% is therefore reasonable.

PUBLICATIONS AND PRESENTATIONS

C. E. Backus, "Laser Activation of Solar Cells," presented at the Ninth IEEE Photovoltaic Specialist Conference, Silver Spring, MD, May 1972.

T. G. Frank and C. E. Backus, "Effect of Neutron Spectra on the Swelling of Ceramic Insulators and Implications for Thermionic Reactor Design."

W. A. Ranken, T. G. Frank, and G. W. Keilholtz, (ORNL); "Effect of Fast Neutron Irradiation on Alumina and Yttria."

W. A. Ranken, and W. H. Reichelt, "Behavior of Tungsten-Clad Mo-UO₂ Fuel Under Neutron Irradiation at High Temperature."

The last three papers were presented at the 3rd International Conference on Thermionic Electrical Power Generation, Jülich, Germany, June 5-9, 1972.