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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 April 30, 1972

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This report presents the status of the Space Electric Power Research and Development program at LASL. The four most recent reports in this series are:

LA-4746-MS (Part I)

LA-4844-MS (Conf. RD)

LA-4747-MS (Part II)(Conf. RD)

LA-4898-PR (Conf. RD)

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QUARTERLY STATUS REPORT ON THE
SPACE ELECTRIC POWER R AND D PROGRAM
For the Period Ending April 30, 1972

SUMMARY

Ceramic-metal multilayers containing Y_2O_3 as the electrical insulator have been fabricated by arc-plasma-spraying Y_2O_3 onto metal collectors, followed by isostatic pressing of the trilayer assembly. Some properties of sprayed Y_2O_3 before and after isostatic pressing have been determined. Arc-plasma-spraying of Y_2O_3 results in a dense, small-grained ceramic of low porosity after isostatic pressing at 10 000 psi and 1550°C. The electrical resistivity of a Nb-1%Zr/ Y_2O_3 trilayer has been accurately measured, and long-term tests at temperature are in progress.

The postirradiation inspection and evaluation of alumina specimens irradiated by Oak Ridge National Laboratory in Capsule ORNL EB-2 is nearing completion. Volume expansions of Lucalox, Coors AD-995, and WESGO Al-995 have been determined. The swelling of Lucalox was generally less than previously observed for similar material. The Coors AD-995 swelled considerably more than Lucalox and was very fragile; and the WESGO Al-995 swelled more than either of the other materials and more than previously observed for similar material. Leak tests indicate very low probability that cylindrical shells will retain vacuum integrity if radiation-induced swelling exceeds 3 vol%.

An isothermal irradiator test unit has been constructed which duplicates cooling conditions in the Experimental Breeder Reactor (EBR-II) better than has been possible in the past. Initial tests of the prototype heat pipe for Isothermal Irradiator K-5 have been completed.

Force-deflection measurements on ceramic bodies indicate decreases in moduli of elasticity and rupture of about a factor of two for irradiated alumina with a volume swelling of 3% compared to unirradiated material. No apparent decreases in these moduli due to irradiation were noted for Y_2O_3 for neutron fluences of $\sim 4.5 \times 10^{21}$ n/cm².

Measurements of thermal diffusivity of one specimen of irradiated AD-999A alumina indicate no change due to irradiation at specimen temperatures above 500°C.

A fracture sample of the tungsten cladding from General Electric Thermionic Test Assembly SCIP-518 was examined with a scanning-electron microscope. Numerous voids (0.2 to 2.5 μ m diam) were detected in the cladding near the fuel/cladding interface. These voids decrease rapidly in size and number with increasing distance from this interface; they occur predominately on grain-boundary surfaces. The locations of the voids could not be correlated with the locations of fission products detected at the fuel/cladding interface.

SHEATHED INSULATOR DEVELOPMENT

Characterization of Multilayers

Much of the current effort in LASL's thermionics program is devoted to the development of ceramic/metal multilayers containing Y_2O_3 as the electrical insulator. This material has insufficient mechanical strength to permit the fabrication of thin-walled cylinders by conventional machining techniques. Therefore, the technique currently being used is to apply the Y_2O_3 to the collector by arc-plasma spraying. Some properties of sprayed Y_2O_3 before and after isostatic pressing at 10 000 psi and 1550°C are listed in Table I.

Photomicrographs of etched Y_2O_3 , as-sprayed and after isostatic pressing are shown in Fig. 1, scanning-electron micrographs are shown in Fig. 2. Clearly, arc-plasma spraying of Y_2O_3 results in a dense, small-grained ceramic of low porosity after isostatic pressing at 10 000 psi and 1550°C. The force necessary to fracture Y_2O_3 in a trilayer is relatively low. There is some indication that damage done during the cutting of the tensile specimens is a contributing factor. As reported previously, the presence of a thin (0.002-in.-thick) layer of Al_2O_3 between the metal and the Y_2O_3 in a

trilayer more than doubles the force required to fracture the ceramic in yttria trilayers (Quarterly Report LA-4898-PR). Studies are in progress to investigate further the effects of Al_2O_3 and other oxide additives on the strength and other properties of Y_2O_3 .

Electrophoretic Deposition of Y_2O_3

Relatively dense, smooth, and uniformly thick (up to 0.007 in.) layers of Y_2O_3 were readily deposited onto Nb-1%Zr collectors from a suspension of Y_2O_3 in isopropanol at electric potentials varying from 500 to 1000 Vdc. The green strength of the deposits probably was adequate to assemble an outer sheath, but severe crazing of the deposit occurred as the alcohol evaporated. The crazing was probably due to the small particle size ($\approx 1 \mu m$) of the Y_2O_3 . Subsequent deposits will be made from suspensions containing larger Y_2O_3 particles.

Electrical Resistivity Tests

Apparatus and procedures capable of reliable electrical resistivity measurements of sheathed insulators, both in and out of pile, are being developed. The apparatus is shown schematically in Fig. 3. Nickel support cones were used for the

TABLE I
PROPERTIES OF ARC-PLASMA SPRAYED Y_2O_3 ⁽¹⁾

	As Sprayed	After Hot Isostatic Pressing ⁽²⁾
Average grain size	--	5 μm (2-16 μm)
Knoop hardness	--	750 (100)
Density ⁽³⁾	4.55 g/cm ³ (91%)	4.91 g/cm ³ (98%)
Interconnected porosity ⁽³⁾	9% (pores 0.12 to 0.6 μm)	< 1% (pores < 0.2 μm)
Specific resistance at 20°C	--	$1.2 \times 10^{17} \Omega cm$
Specific resistance at 750°C	--	1.3 to $4 \times 10^5 \Omega cm$
Tensile strength at 20°C ⁽⁴⁾	--	≈ 800 psi

(1) Arc-plasma parameters: argon gas, 55 psi, 45 cfm; 400 A; sprayed onto 0.563-in.-diam Nb-1% Zr cylinders.

(2) Isostatically pressed in 10 000-psi helium at 1550°C.

(3) Determined with mercury porosimeter; maximum, 15 000 psi.

(4) Pull test on segments cut from a cylindrical trilayer 0.5 in. long by 0.375 in. chord length. Pull rate, 0.010 in./min.

(a)

20 μ m

(b)

20 μ m

Fig. 1. Micrographs of arc-plasma-sprayed Y_2O_3 ; (a) before and (b) after pressure-bonding at 10 000 psi and 1550°C. Etched in hot KOH.

(a)

10 μ m

(b)

10 μ m

Fig. 2. Scanning-electron micrographs of fracture surfaces of arc-plasma sprayed Y_2O_3 ; (a) before and (b) after pressure-bonding at 10 000 psi and 1550°C.

initial test, but at 750°C and $\approx 10^{-7}$ Torr, nickel was deposited onto the exposed ceramic, allowing current to leak to the outer sheath. The support cones have been replaced with molybdenum cones and the test is continuing.

The electrical conductivity of arc-plasma-sprayed Y_2O_3 in a Nb-1%Zr trilayer is shown in Fig. 4. The conductivity of metallized cylinders machined from pressed and sintered Lindsey Co. Y_2O_3 powder is shown for comparison.

NEUTRON-INDUCED DAMAGE TO CERAMICS AND CERAMIC-METAL COMPONENTS

Disassembly of ORNL Irradiation Capsule EB-2

The postirradiation inspection and evaluation of alumina specimens irradiated by Oak Ridge National Laboratory (ORNL) in Capsule ORNL EB-2 is nearing completion. The capsule had been irradiated in Position 7D5 of the EBR-II core during reactor Runs 46A through 50H for a total of 7572 Mwd. Analyses

of the SiC temperature monitors included in the capsule indicate that maximum irradiation temperatures ranged from 815° to 950°C.

Fast-neutron fluence ($E_n > 1.0$ MeV) estimates were based on the number of $^{54}Fe(n,p)^{54}Mn$ reactions in three stainless-steel flux monitors included in the capsule and in five stainless-steel rings cut from the outer containment tube. Conversions of neutron fluences $E_n > 1.0$ MeV, to neutron fluences $E_n > 0.1$ MeV, were based on published analytical results.¹

Volume expansions for Lucalox, Coors AD-995, and WESGO Al-995 samples are plotted vs neutron fluence ($E_n > 0.1$ MeV) in Figs. 5, 6, and 7, respectively. The least-squares fits to previous data for small-grain Lucalox and Coors AD-999 are included for comparison.

The swelling of Lucalox was generally less than previously observed for similar material. The Coors AD-995 swelled considerably more than Lucalox and was very fragile. Three of the five solid cylinders cracked during irradiation. However, the five cylindrical shells were all intact. The WESGO Al-995 swelled more than either of the other

Fig. 3. Schematic of apparatus used to measure the electric resistivity of sheathed insulators.

Fig. 4. Conductivity of arc-plasma-sprayed and isostatically pressed Y_2O_3 .

Fig. 5. Volume expansion of Lucalox in Capsule ORNL EB-2 vs neutron fluence.

Fig. 6. Volume expansion of Coors AD-995 in Capsule ORNL EB-2 vs neutron fluence.

materials and more than previously observed for similar material. These specimens were extremely fragile. One cylindrical shell broke during irradiation and the rest broke while dimensional measurements were being taken. However, the solid WESGO cylinders did not crack during irradiation. The measured volume increases for broken and cracked specimens are less reliable than those for specimens that survived intact.

Twelve of the thirty samples in Capsule ORNL EB-2 were mounted in epoxy and examined metallographically. Typical unetched photomicrographs of WESGO Al-995, Coors AD-995, and Lucalox specimens

Fig. 7. Volume expansion of WESGO Al-995 in Capsule ORNL EB-2 vs neutron fluence.

are shown in Figs. 8, 9, and 10, respectively. Grain-boundary separation was very pronounced for both the Al-995 and AD-995 samples. Considerable grain pull-out occurred during polishing of these materials. The Coors AD-995 swelled asymmetrically: The ratio of fractional increase in length to fractional increase in diameter was ~ 0.65 , but metallographic examinations of cross sections and of longitudinal sections of this material did not reveal any significant differences in microstructure or grain size.

Samples of each of the materials were examined by means of a scanning-electron microscope. Scanning-electron micrographs of WESGO Al-995, Coors AD-995, and Lucalox are shown in Figs. 11, 12, and 13, respectively.

Impurities are readily discernible in the WESGO material (Fig. 11). Examination with an electron-beam microprobe revealed that the material between grains consists primarily of silica and calcia. The voids in the grains of both the WESGO and Coors samples are sintering voids. The Lucalox has much less grain-boundary separation than the other two materials.

Fig. 8. WESGO Al-995 unetched metallographic sample, volume expansion 8.94%.

Fig. 10. Lucalox unetched metallographic sample, volume expansion 3.21%.

Fig. 9. Coors AD-995 unetched metallographic sample, volume expansion 4.84%.

Leak tests were made on all cylindrical shells that were intact after dimensional measurements. Of the five Lucalox cylindrical shells, three were leak-tight and two leaked. These data are plotted vs volume expansion together with previous data in Fig. 14. A volume expansion of ~ 3% appears to be tolerable without loss of vacuum integrity.

Leak tests on Coors AD-995 specimens revealed that two out of three leaked. The leak rates were too large to be measured with a helium-leak detector, but pressurized immersion tests in alcohol did not reveal cracks or holes large enough to form bubbles. The insulator specimens were subsequently partially coated with vacuum grease and again tested with the leak detector. Tests with a very dilute mixture of helium in air led to the conclusion that the leak rates were roughly directly proportional to the non-coated surface area and that leakage was caused by grain-boundary separation.

The moduli of elasticity and rupture have been measured on all cylindrical-shell specimens (including five Lucalox specimens and one of AD-995) which survived dimensional measurements and leak-testing. The results of these measurements are discussed elsewhere in this report (see "Moduli of Elasticity and Rupture," p. 10).

Reference

1. W. Edwards, W. Henderson, and N. Baumgardt, "Analysis of EBR-II Neutron Spectra by Monte Carlo and Discrete Ordinate Methods," General Electric Co., Cincinnati, OH, Nuclear Systems Programs Report GEMP-742 (Dec. 1969).

(a)

20 μ m

(b)

10 μ m

Fig. 11. Scanning electron micrograph of irradiated WESGO Al-995, volume expansion 8.94%.

(a)

20 μ m

(b)

10 μ m

Fig. 12. Scanning electron micrograph of irradiated Coors AD-995, volume expansion 4.84%.

(a) 20 μm

(b) 10 μm

Fig. 13. Scanning electron micrographs of irradiated Lucalox, volume expansion 3.61%.

Fig. 14. Leakage of Lucalox cylindrical shells vs volume expansion.

Isothermal Irradiation Capsules K-3 and K-4

Irradiation Capsules K-3 and K-4 were removed from the Experimental Breeder Reactor (EBR-II) on March 22, 1972, after having been irradiated in Grid Position 7C1 for 6331 Mwd. The peak neutron fluence is estimated to be $\sim 6 \times 10^{21}$ n/cm² ($E_n > 0.1$ MeV). These units contain 87 cylindrical-shell ceramic and ceramic-metal multilayer specimens which were irradiated at temperatures of 450°, 600°, and 750°C.

Isothermal Irradiation Capsules K-5 and K-6; Prototype Tests

In order to provide an isothermal irradiator test unit which duplicates the cooling conditions in the EBR-II much more closely than has been possible in the past, a system has been constructed which utilizes oil cooling at temperatures up to 300°C. The system is shown schematically in Fig. 15 and consists of a pump, a flowmeter, a volume-expansion tank, cooling coils, and an oil-cooled jacket that is clamped tightly to the heat-dump portion of the irradiation-capsule heat pipe. The heat pipe is supported inside a quartz enclosure, and γ -heat is simulated by rf induction heating. Heat removed from the heat pipe by the cooling jacket is dumped by convection from the cooling coils. The heat-removal rate is determined from the oil flow rate and the cooling-jacket temperature rise. The expansion tank is provided with electric strip heaters so that the average oil temperature can be increased if desired. The system is operated with a low overpressure (few psi) of argon supplied at the expansion tank.

Fig. 15. Components of high-temperature, oil-cooled heat-pipe test assembly.

Initial tests of the prototype heat pipe have been completed. Some test results, Fig. 16, are shown as plots of temperature at various axial positions along the heat pipe for different oil-coolant temperatures and for a constant heat input of 655 W to the evaporator section of the heat pipe. The heat pipe is insensitive to heat-dump temperature, and the temperature profiles are consistent with the type of control anticipated. However, the heat-dump section of the heat pipe was not operated at as high a temperature as it will be in the EBR-II. Extrapolation to EBR-II operating conditions indicates that the amount of buffer gas should be somewhat less than that included in the prototype.

The irradiation capsules will be designed for a γ -heat input of ~ 400 W to the evaporator section of the heat pipes; however, there is considerable uncertainty in local γ -heating rates in the EBR-II, and the actual heat input may vary considerably from the anticipated value. The buffer-gas control system should provide operation at essentially constant temperature ($\pm 20^\circ\text{C}$) over a heat-input range from ~ 200 to ~ 800 W. The prototype heat pipe operated satisfactorily at heat inputs from 200 to 700 W. The temperature of the evaporator section of the heat pipe is given as a function of power input to the heat pipe for a relatively low heat-dump temperature in Fig. 17.

The objectives of these tests include the development of a relatively simple testing procedure by which irradiation-capsule heat pipes can be certified for proper operation for this series of EBR-II tests.

Fig. 16. Temperature profiles of prototype isothermal-irradiator heat pipe.

Fig. 17. Temperature of heat-pipe evaporator section vs power input to heat pipe; heat dump section, $\sim 140^\circ\text{C}$.

Moduli of Elasticity and Rupture

Force-Deflection Measurements. A device for force-deflection measurements on ceramic bodies was fabricated and assembled. Measurements are made on C-shaped specimens cut from cylindrical shells. The specimen is subjected to a variable, measured load and its deflection is measured as a function of the magnitude of the load. The load is increased until the specimen breaks; thus, data are obtained that permit the determination of both the modulus of elasticity and of rupture.

Moduli of Al_2O_3 . Values of the moduli of elasticity and of rupture provided by the manufacturers of various types of high-grade Al_2O_3 are listed in Table II together with values measured in the manner described above. The modulus of elasticity is essentially constant for the several types of Al_2O_3 listed, and measured values agree very well with those quoted by the manufacturers. Higher moduli appear to be associated with smaller average grain-size material. This was true for Lucalox for which measurements were made on materials whose average grain size ranged from 10 to 20 μm , as well as for comparisons between Lucalox and AD-999/AVCO materials with grain sizes ranging from 3-5 and 2-3 μm , respectively.

There is considerable variation in both manufacturers' and measured values of modulus of rupture. Again, there is some tendency toward higher moduli for smaller average-grain-size materials.

Moduli of elasticity for Lucalox, AD-999A, AD-999B, and AD-995 are plotted as a function of radiation-induced swelling in Fig. 18. These data

Fig. 18. Modulus of elasticity of Al_2O_3 vs radiation-induced volume swelling.

were obtained from materials irradiated in LASL Irradiation Capsule K-2 and in ORNL Capsule EB-2. Although there is considerable data scatter, the moduli of small-grain Lucalox and AD-999 apparently decrease to about half the unirradiated value at a volume swelling of ~ 3%. This corresponds approximately to the amount of volume swelling at which these materials lose their vacuum integrity.

TABLE II

MODULI OF ELASTICITY AND RUPTURE OF VARIOUS TYPES OF Al_2O_3

Type of Al_2O_3	Manufacturers' Values		Measured Values	
	Elasticity, kgf/mm 2	Rupture, kgf/mm 2	Elasticity, kgf/mm 2	Rupture, kgf/mm 2
Lucalox	4.0×10^4	38-39	$3.4-4.0 \times 10^4$	27-37
AD-999	3.9×10^4	74	$4.2-4.6 \times 10^4$ (999 X)	43-54
			$4.1-4.5 \times 10^4$ (999 B)	42
AVCO	4.2×10^4	42	$4.3-4.6 \times 10^4$	39-41
E-37	3.8×10^4	53	--	--

Fig. 19. Modulus of rupture of Al_2O_3 vs radiation-induced volume swelling.

The single point for AD-995 confirms previous observations that this material has very little strength at a volume swelling of $\sim 3.3\%$. (This was the only specimen of AD-995 and Al-995 which survived to this point in the posttest diagnostic measurements process.)

Values of modulus of rupture are shown in Fig. 19 for the same set of materials as those included in Fig. 18. There are, in general, the same fractional decreases in modulus due to irradiation as had been observed for the modulus of elasticity.

Moduli of Y_2O_3 . Measured moduli of elasticity of various unirradiated Y_2O_3 specimens ranged between one-fourth and one-half the nominal value for unirradiated Al_2O_3 . The highest value was measured for one specimen of Yttralox. Measured values of the modulus of elasticity are plotted versus neutron fluence in Fig. 20. There is no apparent decrease in this modulus due to irradiation, even for specimens which developed hairline cracks and therefore were not vacuum-tight. Two of the three irradiated Y_2O_3 specimens (fabricated from Molybdenum Corp. of America powder) leaked. Note that the maximum neutron fluence for which data are available is $\sim 4.5 \times 10^{21}$ n/cm 2 .

Fig. 20. Modulus of elasticity of irradiated Y_2O_3 vs neutron fluence.

Measured values of the modulus of rupture for Y_2O_3 are shown in Fig. 21. Except for one anomalously high value for an irradiated specimen, there is no apparent effect on this modulus due to irradiation.

Thermal-Diffusivity Measurements

An opaque platinum coating, applied to one side of ceramic specimens for thermal-diffusivity measurements by laser-flash techniques, has been developed successfully. Adequate coatings are obtained by precipitating the metal from platinum chloride during baking of the specimen. Both the baking temperatures and the baking durations should be minimized to prevent possible annealing of irradiated specimens. The minimum plating temperature has been determined experimentally to be 500°C for a baking period of 15 min in vacuum.

Thermal-diffusivity measurements have been made on several unirradiated control specimens of the materials included in Irradiation Capsule K-2. These data have been converted to thermal-conductivity values by using published values of specific heat¹ and are plotted versus temperature in Fig. 22. Although considerable variation in thermal conductivity

Fig. 21. Modulus of rupture for irradiated Y_2O_3 .

exists for the various types of alumina at room temperature, there is only ~ 25% dispersion in the measured values at thermionic operating temperatures (~ 750°C).

Measurements made for one sample of Y_2O_3 (fabricated from Molybdenum Corp. of America powder) are plotted in Fig. 23. These measurements agree very well with published data¹ at low temperatures, but are ~ 30% higher at 750°C.

The thermal diffusivity of one specimen of AD-999A, irradiated to a neutron fluence of 3.3×10^{21} n/cm² ($E_n > 0.1$ MeV) in Irradiation Capsule K-2, has been measured. The room-temperature value was 59% of that of unirradiated material, but there was no difference between measured values for unirradiated and irradiated materials above 500°C. After heating for 2 h at 750°C, the room-temperature diffusivity of the irradiated material had increased to 74% of the value for unirradiated material.

Thermal conductivities of unirradiated and irradiated AD-999A are compared in Fig. 24. It was assumed that specific heat is not affected by irradiation.

Reference

1. Y. S. Touloukian, Ed., "Thermophysical Properties of High-Temperature Solid Materials," 4, MacMillan Co., 1967.

Fig. 22. Thermal conductivity of various types of high-purity Al_2O_3 vs temperature.

FUELED EMITTER DEVELOPMENT

Diagnostic Inspection of GE Thermionic Test Assemblies

A fracture sample of the tungsten cladding from GE Thermionic Test Assembly SCIP-518 was examined with a scanning-electron microscope. An overall view displaying the fracture mode and grain structure from edge to edge is shown in Fig. 25. A narrow band of small equiaxed grains can be seen along the fuel side of the cladding, with large columnar grains running to the outside cladding surface. The fracture mode appears to be predominately intergranular.

Numerous small voids were detected in the cladding near the fuel/cladding interface (see Fig. 26). These voids, ranging in diameter from 0.2 to 2.5 μ m, decrease rapidly in size and number with increasing distance from the fuel/cladding interface. They occur predominately on grain-boundary surfaces and are not evident within the transgranularly fractured grains, one of which is shown in Fig. 27.

Fig. 23. Thermal conductivity of Y_2O_3 vs temperature.

Fig. 24. Thermal conductivity of unirradiated and irradiated AD-999A.

Fig. 25. Scanning electron micrograph of fuel cladding from GE Thermionic Test SCIP-518. Fuel side is at right.

Fig. 26. Scanning electron micrographs of fuel cladding near fuel/cladding interface from GE Thermionic Test SCIP-518.

The sample was subsequently examined with an electron-beam microprobe in an attempt to identify fission products that might have been deposited in the cladding. Molybdenum and technetium were observed in the tungsten immediately adjacent to the fuel/cladding interface but not elsewhere in the cladding. The molybdenum and technetium concentrations could not be correlated with the locations of the voids; zirconium, cesium, iodine, and praseodymium were not detected. Detection limits were estimated to range between 0.1 and 1.0%.

Status of ETR Irradiation Tests

The 12.2-mm-diam Mo-9%W-UO₂ cermet fuel specimens currently being irradiated in the Engineering Test Reactor (ETR) had accumulated 3041 equivalent full-power hours on April 30, 1972. We have been informed that the ETR is scheduled to cease operation at the end of Calendar Year 1972. The reactor is presently being operated ~ 45% of the time; at this rate, the cermet fuel specimens will accumulate ~ 5000 h of exposure by the time of final shutdown.

10 μm

Fig. 27. Scanning electron micrograph of transgranular grain fracture in fuel cladding from GE Thermionic Test SCIP-518.

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