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Quarterly Status Report on the

Space Electric Power R and D Program (U)

for the Period Ending October 31, 1971

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los alamos
scientific laboratory
of the University of California
LOS ALAMOS, NEW MEXICO 87544

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QUARTERLY STATUS REPORT ON THE
SPACE ELECTRIC POWER R AND D PROGRAM (U)
For the Period Ending October 31, 1971

SUMMARY

The neutron-damage model that had been used to correlate radiation-induced swelling of Al_2O_3 has been extended to Y_2O_3 and MgO . Values of the neutron-damage parameter were calculated for each material as a function of neutron energy, and a threshold neutron energy was determined which can be used for all three materials to define a damage neutron fluence with a maximum error of 15%.

Postirradiation measurements of vacuum integrity have been completed for the ceramic specimens and ceramic-to-metal seals included in EBR-II Irradiation Capsule K-2. Leaks were found in two of three Y_2O_3 specimens fabricated from material obtained from the Molybdenum Corporation of America. Two ceramic-to-metal seals were also not vacuum-tight; both had been fabricated by brazing.

Several specimens of autoclave-pressure-bonded trilayers and pentallayers, which are being irradiated in the EBR-II, have been characterized with respect to ceramic grain size and metal-ceramic bond strength for comparison with postirradiation values.

Determinations of fuel burnup and of the fraction of fission gases released from the fuel have been completed for General Electric thermionic-converter Test Unit SCIP-518. The fuel burnup was 2.8×10^{20} fissions/cm³ of emitter interior volume, and 92% of the fission gases were released from the fuel.

Measurements of performance improvement in heat pipes after removal of impurities from the working fluid have shown that impurities in sodium stainless-steel heat pipes have a deleterious effect of similar magnitude as in lithium Nb-1%Zr heat pipes.

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NEUTRON-INDUCED DAMAGE TO INSULATING MATERIALS

Neutron Damage Parameters for Yttria and Magnesia

The neutron-damage model that had been used to correlate radiation-induced swelling of Al_2O_3 (see Quarterly Status Report LA-4497-MS, August, 1970) has been extended to Y_2O_3 and MgO .

The model is based on the assumption that neutron damage is proportional to the number of atoms displaced in the irradiated material. The constants of proportionality are not determined, but, rather, estimates are made of the dependence on neutron energy of the number of displacements resulting from a neutron interaction. The damage model can thus be used to compare the neutron-damage data from reactors with different neutron spectra.

The damage parameter for Y_2O_3 is defined as

$$\begin{aligned} \text{Damage Parameter} &= \int_0^{\infty} K_1 [\sigma_s^Y(E) v_Y(E) + \\ &\quad + 3\sigma_s^O(E) v_O(E)] \phi(E) dE = \\ &= \int_0^{\infty} (RD)_{\text{Y}_2\text{O}_3} \phi(E) dE \end{aligned}$$

and for MgO ,

$$\begin{aligned} \text{Damage Parameter} &= \int_0^{\infty} K_1 [\sigma_s^{\text{Mg}}(E) v_{\text{Mg}}(E) + \\ &\quad + \sigma_s^O(E) v_O(E)] \phi(E) dE = \\ &= \int_0^{\infty} (RD)_{\text{MgO}} \phi(E) dE \end{aligned}$$

where

$(RD) = f(E) =$ relative displacements,

$K_1 =$ arbitrary constant = 0.001 (same as for Al_2O_3),

$\sigma_s(E) =$ microscopic neutron-scattering cross sections obtained from BNL-325 and its Supplements (Brookhaven National Laboratory, Neutron Cross Sections, Second Edition, 1958),

$v(E) =$ number of atomic displacements created per neutron interaction, and

$\phi(E) =$ neutron flux normalized so that $\int_0^{\infty} \phi(E) dE = 1.0$ MeV

The quantity $v(E)$ is a function of E_p , the energy of the initial displaced atom (primary knock-on atom) resulting from a neutron-atom collision. The relationship between the incident neutron energy, E_n , and E_p is

$$E_p = \frac{2M}{(1+M)^2} E_n$$

where M is the mass of the primary knock-on atom.

Thus

$$E_p^O = 0.1107 E_n,$$

$$E_p^Y = 0.002200 E_n, \text{ and}$$

$$E_p^{\text{Mg}} = 0.07593 E_n.$$

One additional quantity is required by the formulation of the model, i.e., the threshold energy, E_I , where ionization of the target material is caused by neutron interactions and the number of displacements per interaction becomes constant. The threshold energy is given by:

$$E_I = \frac{M}{m_e} \left(-\frac{I}{6} \right)$$

where

$m_e =$ mass of electron, and

$I =$ forbidden-energy gap for the target material, assumed to be 10 eV for the materials considered.

The factor 6 in the preceding equation was determined empirically from previous correlations of Al_2O_3 irradiation data. Values of E_I are then

$$E_I^Y = 0.272627 \text{ MeV},$$

$$E_I^O = 0.049067 \text{ MeV, and}$$

$$E_I^{\text{Mg}} = 0.074520 \text{ MeV.}$$

The number of displacements resulting from a neutron-atom collision is assumed to be given by

$$v(E_p) = \begin{cases} 0, & E_p < 25 \text{ eV} \\ 1, & 25 \text{ eV} \leq E_p < 50 \text{ eV} \\ E_p/50, & 50 \leq E_p \leq E_I \\ E_I/50, & E_I < E_p. \end{cases}$$

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From values of E_T , given above, the neutron energies, E_n , at which the number of displacements per neutron interaction becomes constant can be determined. Values of E_n and of the maximum number of displacements per neutron interaction with the respective atoms are

Element	E_n , MeV	Maximum Number of Displacements per Interaction
O	0.443	981
Y	12.4	5453
Mg	0.981	1490

The value of $v^Y(E)$ increases linearly with neutron energy for all neutron energies of interest ($E_n < 10$ MeV). The value of $(RD)_{Y_2O_3}$ exhibits similar behavior and was estimated, for neutron energies greater than 1.5 MeV, by using

$$(RD)_{Y_2O_3} = 8.085 + 1.7435 E.$$

Values of (RD) have been computed for Y_2O_3 and MgO and are shown, together with values of $(RD)_{Al_2O_3}$, in Fig. 1.

It was also of interest to attempt to determine threshold neutron energies for Y_2O_3 and MgO that could be used to define a damage neutron fluence. The analysis was carried out in the same manner as previously done for Al_2O_3 . Values of the quantity C , given by

$$C = \frac{\int_0^\infty (RD) \phi(E) dE}{\int_{E_T}^\infty \phi(E) dE}$$

where $\int_{1.0 \text{ MeV}}^\infty \phi(E) dE = 1$ and E_T was varied from 0.5 to 1.0 MeV,

were calculated for Y_2O_3 and MgO for several different reactor neutron spectra.

If a value of E_T can be found that results in the same value of C for various neutron spectra, then the numbers of atomic displacements predicted by the neutron damage model in different neutron spectra will be proportional to neutron fluence, $E_n > E_T$.

The results of these calculations are given in Figs. 2 and 3 for Y_2O_3 and MgO, respectively. The Y_2O_3 data converge at a value of $E_T \sim 0.15$ MeV, which is the same value as that for Al_2O_3 . The MgO data converge below 0.1 MeV. However, the variation in the value of C at 0.1 MeV is less than 15% for MgO, Y_2O_3 , and Al_2O_3 ; therefore, neutron fluence, $E_n > 0.1$ MeV, can be used to compare the damage potential of various neutron spectra for all three materials in practical applications without incurring an error greater than 15%.

Fig. 1. Displacements per unit of neutron flux vs neutron energy.

Fig. 2. Ratio of damage parameter for Y_2O_3 to integrated neutron flux vs lower limit of integration.

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Fig. 3. Ratio of damage parameter for MgO to integrated neutron flux vs lower limit of integration.

Insulator Irradiation Capsule K-2

Measurements of vacuum integrity were made for all test specimens that had been included in EBR-II Irradiation Capsule K-2 (see Quarterly Status Report LA-4747-MS, August 1971). Each specimen was tested with a helium-leak detector and was submitted to a bubble test if a leak was indicated, to determine the nature of the leak.

The only cylindrical shell insulators that were not vacuum-tight were two of the three Y_2O_3 specimens which had been fabricated from Y_2O_3 powder obtained from the Molybdenum Corporation of America (MCA). All three insulators had a mottled appearance, as shown in Fig. 4. The leaks resulted from very fine, single hairline cracks with axial orientation. These cracks may have occurred during posttest handling rather than during irradiation. In any event, the cracking indicated the fragility of the specimens. It is uncertain whether the cracking is directly related to irradiation exposure because the uncracked specimen had been exposed to a neutron fluence 25 to 50% higher than the two cracked units.

Two Y_2O_3 insulators fabricated from powder supplied by Lindsey Co., were vacuum-tight. Fabrication of leak-tight specimens from material supplied by MCA was much more difficult than from the Lindsey powder, and the former specimens were probably structurally weaker. Rupture tests will be performed on both irradiated and unirradiated specimens to verify this supposition.

Fig. 4. Cylindrical-shell Y_2O_3 insulat after irradiation.

Two insulator-seal assemblies were not vacuum-tight; both had been fabricated from Nb/Al-995 and had been brazed with Nb-V eutectic alloy. Tungsten had been applied to the ceramic by use of the Gulf Energy and Environmental Systems, Inc. (GEES) modification of the IASL metallizing technique, i.e., a 14-to-16- μ m thick tungsten layer was deposited by chemical vapor deposition (CVD) to seal the voids in the base layer which had been produced by high-firing a W- Y_2O_3 slurry. One assembly is shown during testing in Figs. 5 and 6. Numerous cracks are easily discernible. Leakage in both assemblies occurred through a small pin hole at the edge of the tungsten metallized layer. There is essentially no doubt that the leaks occurred in this metallizing layer rather than in the ceramic.

All hot-gas pressure-bonded insulator seals included in Capsule K-2 were vacuum-tight, including one seal fabricated at IASL from Nb-1%Zr and Feldmühle E-37 alumina. Also, the flat seal which was provided by the Brown, Boveri & Cie (BBC) thermionics group and which consisted of Ni/E-37 brazed with Cu-1%Ni alloy was leak-tight. Both seals containing E-37 had been exposed to the maximum neutron fluence of $\sim 4.5 \times 10^{21}$ nvt ($E_n > 0.1$ MeV). These two seals are shown in Figs. 7 and 8.

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Fig. 5. Braze insulator seal assembly; note cracks in metallizing layer.

Fig. 6. Braze insulator seal assembly during bubble test.

Fig. 7. Ni/E-37 insulator seal assembly after irradiation.

Fig. 8. Hot-gas pressure-bonded Nb-1%Zr/E-37 insulator seal assembly after irradiation.

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The insulator seals were cut axially to allow metallographic examination of the metal-ceramic interfaces. This procedure produced separation of one or both of the metal bonds from the ceramic substrate for every sample except the copper-brazed BBC seal. The cutting procedure resulted in severe vibration of the seals and mounting structure; however, destruction of the seals was not anticipated, which emphasizes the need for investigation of the effects of irradiation on the strengths of metal-ceramic bonds.

SHEATHED-INSULATOR DEVELOPMENT

Characterization of Multilayers

Selected specimens of autoclave-pressure-bonded trilayers and pentallayers which are being irradiated in the EBR-II (Irradiation Capsules K-3, K-4) have been characterized with respect to ceramic grain size and metal-ceramic bond strength in addition to the usual measurements to determine dimensions and vacuum integrity. Measurements of ceramic grain size and bond strength were made on specimens which were cut from the same tri- or pentallayers from which the test specimens were obtained.

Tensile data, listed in Table I, were obtained on 0.5-in.-wide circular segments having a chord length of 0.375 in.; the samples had been brazed at 850°C in vacuum with BT solder, using titanium flux. The pull rate was 0.010 in./min at room temperature.

Note that the bond strength of Specimen SDPL-121, which had been formed by spraying both ceramic layers as well as the metal intersheath, was as great as any measured in this series. Specimen SDPL-96, which had been fabricated by pressure-bonding machined cylinders of Al_2O_3 and Nb-1%Zr, separated at 14% less load (see Table II) than the sprayed pentallayer. Photomicrographs of the two pentallayers are shown in Figs. 9 and 10, respectively. Note that the interfaces formed by arc-spraying are more irregular than for the machined cylinders, which may account for the stronger bond. Oxide inclusions are apparent in the sprayed interlayer of the Nb-1%Zr in SDPL-121. Hardness values, taken at the points indicated in Fig. 9, are listed in Table II. The Nb-1%Zr interlayer, sprayed in air, is considerably harder than the Nb-1%Zr in the nonsprayed metal, indicating a greater oxygen content in the sprayed metal. These data also indicate that oxygen has diffused from the Al_2O_3 into both the collector and the outer sheath. Diamond-point hardness (DPH) values of 104 to 115 are typical of Nb-1%Zr after pressure bonding.

A specimen from SDTL-117 (with a Nb-1%Zr collector, Yttralox ceramic, and Nb-1%Zr outer sheath, see Table III) fractured after brazing during normal handling. The fracture occurred in the body of the ceramic and not at a metal-ceramic interface. Examination of other portions of this trilayer specimen indicated that the Yttralox had cracked severely sometime during the autoclave-machining procedure. Such cracking did not occur in trilayers fabricated from pure yttria.

TABLE I

TENSILE BOND STRENGTH OF MULTILAYERED SHEATHED INSULATORS

<u>Specimen^a</u>	<u>Equivalent Sample in Capsules K-3, K-4</u>	<u>Ceramic</u>	<u>Load at Failure, psi</u>	<u>Point of Failure</u>
SDPL-96	No	Al-995	4783	Collector- Al_2O_3 interface
SDTL-106	Yes	Avco	2019	Outer sheath- Al_2O_3 interface
SDTL-110	Yes	AD-999B	5362	Outer sheath- Al_2O_3 interface
SDTL-104	Yes	AD-999B	990	Outer sheath- Al_2O_3 interface
LDTL-120	No	Al-995	5349	Outer sheath- Al_2O_3 interface
SDPL-121	No	Al_2O_3	5549	Outer sheath- Al_2O_3 interface

^aSD, LD, TL, PL = small- (~ 0.6 in.) or large- (~ 1.3 in.) diameter, tri- or pentallayer.

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200μ

Fig. 9. Hot-gas pressure-bonded Nb-1%Zr/Al₂O₃ pentalayer - Al₂O₃ and metal intersheath are arc-sprayed material. Regions from top: outer sheath, Al₂O₃, metal intersheath, Al₂O₃, collector.

500μ

Fig. 10. Hot-gas pressure-bonded Nb-1%Zr/Al₂O₃ pentalayer formed from machined cylinders. Regions from top: outer sheath, Al₂O₃, metal intersheath, Al₂O₃, collector

from all multilayers listed in Table III, except from SDTL-108, were included in Capsules K-3 and K-4.

Fabrication of Multilayers

The fabrication details of four sheathed insulators which have been pressure-bonded during this report period are shown in Table IV.

The Al₂O₃ and the Y₂O₃ in Samples LDPL-124 and SDTL-128 were applied by plasma-arc spraying. The Al₂O₃ was Norton 500-F, with a grain size of ~ 15μm and the Y₂O₃ was a product of Moly Corp., screened to a particle size of ~ + 40μm. The metal sheath in LDPL-124 had been formed from 0.005-in.-thick Nb-1%Zr sheet with a spot-welded lap joint.

No distortion occurred during pressure-bonding, and ultrasonic scanning indicated good bonding with no voids. One-half-inch-long cylinders cut from the insulators were leak-tight as indicated by a helium-leak detector.

TABLE II

DPH VALUES OF METAL LAYERS IN SDPL-121

<u>Location</u>	<u>DPH</u>
Outer sheath	115
	145
	170
Interlayer	460, 420, 405
Collector	150
	108
	104

The grain sizes of the ceramic in multilayers before and after pressure-bonding are listed in Table III, and will be compared to grain sizes in equivalent specimens after their removal from the EBR-II. Tentative values for the electrical resistivity of these specimens are also listed. These data were obtained at ambient temperature from unguarded 0.5-in.-long specimens. Specimens

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TABLE III
GRAIN SIZES AND ELECTRICAL RESISTIVITIES
OF CERAMIC IN TRILAYERS

No.	Ceramic	Grain Size, μm		Electrical Resistivity, $\Omega\text{-cm}$
		Before Bonding	After Bonding	
SDTL-101	Lucalox ^a	10	27	-
102	"	10	32	7×10^{14}
103	"	10	32	-
104	"	10	27	11×10^{14}
110	AD-999B ^b	3 to 10	16	8×10^{14}
111	"	3 to 10	19	11×10^{14}
105	AD-999X ^b	2 to 3	11	-
106	Avco ^c	2 to 3	8	11×10^{14}
114	"	2 to 3	8	7×10^{14}
108	Y_2O_3 ^d	?	5	-
116	Yttralox ^e	?	76	5×10^9
117	"	?	19	-

^aGeneral Electric Al_2O_3 cylinders

^d Y_2O_3 powder from Lindsey, pressed and machined at LASL

^bCoors Al_2O_3 cylinders

^eGeneral Electric Y_2O_3 cylinders

^cAvco Al_2O_3 cylinders

TABLE IV

Voltage-at-Temperature Irradiation Test of Trilayer Units

INSULATORS PRESSURE-BONDED AT 10,000 psi, 1550°C

Specimen	Composition and Thickness (in.)				
SDTL-123	Nb-1%Zr/AD-999X/Nb-1%Zr ^a 0.035 0.020 0.012				
LDPL-124	Nb-1%Zr/ Al_2O_3 /Nb-1%Zr/ Al_2O_3 /Nb-1%Zr 0.035 0.010 0.005 0.010 0.015				
LDTL-127	Nb-1%Zr/Al-995/Nb-1%Zr 0.035 0.020 0.015				
SDTL-128	Nb-1%Zr/ Al_2O_3 / Y_2O_3 / Al_2O_3 /Nb-1%Zr 0.035 0.003 0.020 0.003 0.012				

^aCollector/ceramic/outer sheath.

The in-pile voltage-at-temperature test assembly which was described in a previous quarterly status report (LA-4625-MS, February 1971), was removed from the Omega West Reactor in June 1971 after 3150 h of irradiation at 750°C. This test unit contained a Nb-1%Zr-Lucalox and a Nb-1%Zr-Al 995 trilayer unit. The outer sheath of each unit was machined to form three Nb-1%Zr bands so that guard-ring measurements of resistivity could be made.

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Posttest radiography and disassembly revealed that the ceramic assembly supporting the electrical leads and connections to the test specimens had been displaced ~ 0.5 in. downward and that all electrical connections to the trilayers had been broken. There was also a conductive layer on the ceramic between the bands, which, according to qualitative chemical analysis, consisted primarily of nickel with some niobium. The source of the nickel was a shield that had been placed around the test-specimen section to minimize the effects of thermionic emission.

All data obtained from this test are suspect. Extensive redesign of the test unit is obviously necessary before similar measurements are attempted on AD-999 alumina and yttria.

FUELED-EMITTER DEVELOPMENT

Disassembly of Thermionic Converters Tested by General Electric Co. (GE)

Measurements of uranium isotopic ratios in the fuel of Thermionic-Converter Test Unit SCIP-518 have been completed. Estimates of fuel burnup were made by comparing pre- and posttest values of isotopic ratios. The fission gases released by dissolution of fuel samples from SCIP-518 have been counted and the fraction of fission gas retained in the fuel

has been determined. These data together with time histories of the test enable reconstruction of the operating conditions of the test unit. The operating conditions, as determined from the data referred to above, are given in Table V.

Note that the actual thermal-power level of this test unit was apparently only ~ 90% of the value predicted by GE.

The fractional release of fission gas from the fuel (92%) was determined from the fission-gas pressure measured in the test assembly and, independently, from the amount of fission gas released by dissolution of part of the fuel. These two determinations agreed to within 1%.

The estimates of cell efficiency given in Table V were based on the assumption that the test unit was in a constant neutron flux throughout the test period, and thus, that the power output was ~ 8% less at the end of the test period than at the beginning because of fuel burnup.

HEAT PIPE SYSTEMS

Introduction

Because of its brevity, the unclassified section on heat pipe systems, normally published separately, has been combined with the remainder of the Space Electric Power R and D Program Quarterly Status Report.

TABLE V
SCIP-518 OPERATING CONDITIONS

Average input power: 383 W fission and 42 W γ -heat (425 W total)^a
220 W/cm³ fission-averaged over emitter interior volume
Average fuel burnup: 2.8 x 10²⁰ fissions/cm³ of emitter interior volume
(4.89 x 10²⁰ fissions/cm³ of full-density fuel volume)
Gap Width: 0.007 in.

Operating time, h	Output Power (Electrode), W	Efficiency (η), % ^b
0	58.2 (6.8 W/cm ²)	13.5
2000	45.8	10.5 ^c
4000	40.3	9.4 ^d
10,178	37.5	9.2

^a470 W predicted
^bUncorrected electrode efficiency
^cVoltage reduction
^dVoltage increase, fission gas in diode

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Investigation of Heat Pipe Purification Methods

Recent investigations at LASL indicate that many problems encountered with alkali metals in heat pipes may be caused by low-volatility impurities collecting in the heated section. As described earlier (see Quarterly Status Report LA-4746-MS, August 1971) a Nb-lw/o Zr heat pipe with lithium as a working fluid could not be started successfully with impurities present. However, once the impurities were removed with an yttrium getter, the predicted heat-transfer capability of the pipe was achieved without difficulty. The same result was obtained with a second lithium heat pipe that had been purified with yttrium.

Further verification that heat transfer can be substantially improved by removing the impurities has been obtained with a stainless-steel heat pipe

containing sodium. After this heat pipe had been operated for several hours, the heated section was cut off and a new heat pipe was formed from the remaining section. All of the wick in the removed heated section was discolored, and part of it was quite brittle. Before removal of this section, the maximum heat-transfer capability of the pipe was 1.5 kW. Since removal, a heat-transfer rate of 7 kW has been achieved, but the maximum capability has not been reached because of equipment limitations.

In current tests with sodium in a stainless-steel heat pipe obtained from Jet Propulsion Laboratory, performance has been somewhat improved by removing impurities with a zirconium getter. However, greater improvement has been achieved by cold-trapping (see LA-4746-MS). As a result of these tests, investigations are now being made to identify the impurities that collect in various alkali-metal heat pipes and to develop satisfactory methods for their removal.

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