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ORNL ISOTOPIC POWER FUELS
QUARTERLY REPORT
FOR PERIOD ENDING DECEMBER 31, 1972

Eugene Lamb

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ISOTOPES DEVELOPMENT CENTER

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Isotopes Division

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SUMMARY

Materials for testing with $^{244}\text{Cm}_2\text{O}_3$ at 900, 1100, and 1400°C are being procured. Welding of the inner and outer compatibility capsules is expected to be completed by March 1, 1973. The metals and alloys tested with $^{244}\text{Cm}_2\text{O}_3$ for 1,000 and 10,000 hr at 750°C were examined metallographically; the results indicated good compatibility. The potential ceramics chosen for a compatibility study with Cm_2O_3 are BeO, SiC, ZrO_2 , and ThO_2 . Preliminary studies of BeO and SiC showed the formation of low-melting phases, which resulted in the rejection of these two ceramics.

Cells in the source fabrication facility have been repaired to provide the proper inert atmosphere for encapsulation of the compatibility samples.

Neodymium oxide is being used as a stand-in for curium oxide during the adjustment and check-out of the mass spectrometer to increase the signal-to-background ratio and thus to increase the sensitivity of mass measurements at low specimen temperatures.

Apparatus is being designed for the measurement of helium release rates from $^{244}\text{Cm}_2\text{O}_3$ pellets in the temperature range 500-1000°C. These data will provide new information on fuel form behavior in power conversion devices designed for lower temperature operation.

The πMC particle size analyzer has been received and calibrated. With proper sample preparation, the instrument is capable of giving size distribution down to 0.1 μm .

CURIUM-244 FUEL DEVELOPMENT

(Division of Space Nuclear Systems Program 04 30 05 03)

 $^{244}\text{Cm}_2\text{O}_3$ Compatibility Program

Couple Component Procurement

Procurement of materials for testing with $^{244}\text{Cm}_2\text{O}_3$ at 900, 1100, and 1400°C is continuing. All of the materials except iridium and Pt_3Ir have either been received or are currently being machined. Iridium of suitable purity has recently been obtained for fabrication into capsules.

The two ceramic materials selected for inclusion in this program are ThO_2 and ZrO_2 . Rods of ThO_2 were prepared and are being machined into modified inner-capsule test-system components. An order has recently been placed to commercially obtain ZrO_2 inner-capsule components.

All graphite intermediate assemblies (35) and the Al_2O_3 capsule holders have been completed and received. The BeO capsule holders have been received from Coors. The inner capsule assemblies have been completed with the exception of the W-Re, Mo-Re and Pt alloys, and the iridium metal. Problems were encountered in fabricating crack-free capsule bodies from the W-Re and Mo-Re alloys, and these are being stress-relieved to avoid further cracking. Machining of capsules from iridium is estimated to be completed by February 15, 1973. The ThO_2 inner capsules are being machined at ORNL, and the ZrO_2 capsules have been ordered from Zircoa Company.

Weld development on the inner capsules is scheduled to start early in January; however, weld development on the iridium capsules cannot begin until the components are available about February 15. Based on these considerations, we expect to have welding completed on all inner capsules and the ten outer capsules by March 1, 1973.

Compatibility Furnace Installation

Five furnaces are required for the three temperatures of exposure (900, 1100, and 1400°C) and for the number of samples to be exposed. Two Brew vacuum furnaces previously used in the curium compatibility program are available. A furnace previously used for $^{90}\text{SrTiO}_3$ compatibility tests has been transferred to FPDL for installation in cell 17 for the 900°C exposure. A surplus Brew vacuum furnace has been obtained from a completed AEC program at Sanders Nuclear Corporation, and a high-temperature TEECO furnace, previously used at ORNL, has been obtained.

The recently acquired Brew vacuum furnace was installed, and service connections are being made. One of the Brew furnaces previously used in the curium compatibility program and a TEECO furnace were moved to permit efficient utilization of the neutron shielding material.

The inclusion of vented couples in this compatibility program requires the installation of inert atmosphere glove boxes on all four high temperature furnaces; this has not been required previously because only sealed couples have been exposed. The number of couples to be exposed simultaneously is much greater than for previous compatibility runs; therefore, the neutron radiation dose rate in the vicinity of the furnaces will require the installation of neutron shielding rather than maintenance of an exclusion area as previously practiced. The neutron shielding will consist of polyvinyl chloride blocks cut from material obtained on surplus from an Oak Ridge installation. The shop fabrication of inert atmosphere glove boxes is complete, and field installation on the four high-temperature furnaces is in progress. The polyvinyl chloride neutron shielding is being cut into sections in preparation for installation around the furnaces. The target date for completion of the installation is mid-February.

Metallographic Examination of Couples Tested at 750°C

The following metals and alloys have been evaluated with $^{244}\text{Cm}_2\text{O}_3$ for 1,000 and 10,000 hr at 750°C: Ni, Ni-10% Cr, Ni-7% Cr-16% Mo-5% Fe (Hastelloy N), Ni-20% Cr, Ni-16% Cr-16% Mo-6% Fe-3% W (Hastelloy C), Co-10% Cr, and Co-20% Cr-15% W-10% Ni (Haynes alloy No. 25). The pure metal nickel and the binary alloys Ni-10% Cr, Ni-20% Cr, and Co-10% Cr were included primarily to obtain base-line information against which we could compare the more complex commercial alloys.

In general, the results indicated good compatibility between $^{244}\text{Cm}_2\text{O}_3$ and all of the materials tested. Metallographic observations are summarized in Table 1 and Figs. 1 and 2. The results with nickel after 1000 hr

Table 1. Summary of Metallographic Examination of Materials Exposed to $^{244}\text{Cm}_2\text{O}_3$ at 750°C

Material	1,000-hr Test	10,000 Test
Nickel	Heavy porosity in grain boundaries of specimen, end plug, and capsule wall to maximum depth of 25 mils	Specimen unaffected; end plug had heavy precipitation or void formation to 0.5 mil
Ni-10% Cr	Surface reaction zone to maximum depth of 0.5 mil. Slight surface roughening	Surface reaction zone to maximum depth of 0.5 mil. Some large voids in reaction zone
Ni-20% Cr	Surface reaction zone to maximum depth of 0.5 mil. Slight surface roughening	Surface reaction zone to maximum depth of 0.5 mil. Surface roughening
Hastelloy N	Unaffected	Surface reaction zone to maximum depth of 0.1 mil
Hastelloy C	Surface roughening	Unaffected
Co-10% Cr	Surface roughening	Surface roughening and slight attack to maximum depth of <0.5 mil
Haynes 25	Very slight surface roughening	Unaffected

Fig. 1. Super Alloys Exposed to $^{244}\text{Cm}_2\text{O}_3$ for 1,000 hr at 750°C. (Samples are unetched.)

Fig. 2. Super Alloys Exposed to $^{244}\text{Cm}_2\text{O}_3$ for 10,000 hr at 750°C. (Samples have been etched.)

probably do not represent reaction with $^{244}\text{Cm}_2\text{O}_3$. Pure nickel has been observed to show porosity, especially after welding, due to the evolution of trapped gases. Since similar results were not found after the 10,000-hr exposure, this seems the more likely explanation. Binary alloys with chromium were consistent in showing some slight attack. We initially

felt that chromium would probably be one of the more active elements in the commercial alloys. Although this would seem verified by the binary alloys, only Hastelloy N of the three commercial alloys showed any evidence of attack after 10,000 hr.

In view of these excellent results, any of the commercial alloys would appear adequate to contain $^{244}\text{Cm}_2\text{O}_3$ at 750°C.

Ceramics-Curia Reactions

The inclusion of ceramic materials in the matrix of the planned study of the compatibility of Cm_2O_3 with containment materials is being considered. Ceramic oxides offer possible advantages as source containment materials, especially their oxidation resistance. From a previous literature study, four ceramics were chosen for preliminary screening tests. These ceramics were BeO , SiC , ZrO_2 , and ThO_2 .

A short-term compatibility experiment was performed with SiC and CmO_2 . Curium dioxide and SiC were weighed out to mole ratios of 1:2, respectively. The powders were mixed by mortar and pestle and then cold-pressed into a pellet. The pellet was loaded into a tungsten crucible, which was put into a high-temperature vacuum furnace. The pellet was observed as the temperature of the furnace was slowly increased. A noticeable increase in gas pressure occurred above 1200°C; at 1560°C, the pellet appeared to be melting. After the pellet was removed from the furnace, it was observed that approximately one-half had melted. The mass fraction lost by the crucible and pellet was 0.345. The melted substance appeared to have a shiny, silvery metallic luster. Part of the unmelted substance was submitted for x-ray diffraction analysis.

Two of the possible reactions which could have occurred are:

The mass fractions lost in reactions (1) and (2) are 0.247 and 0.157, respectively. In the present case more mass was lost than either of the two above reactions could account for. Part of the sample had either volatilized or escaped the crucible. From the increase in gas pressure and from the melting point of the product, it appears as if a curium silicide product was formed. This experiment clearly showed that the material SiC should not be used for containment of Cm_2O_3 at high temperatures.

Some of the product from the SiC and CmO_2 reaction was examined by x-ray diffraction analysis. The lines in the x-ray pattern were rather weak; however, the lines were isoparametric with those of GdSi_2 , indicating that CmSi_2 was formed.

An examination of the $\text{BeO}-^{244}\text{Cm}_2\text{O}_3$ system showed the formation of low-melting eutectics ($\sim 1650^\circ\text{C}$), which was cause for elimination of BeO ceramic from the test matrix.¹

We have completed the screening tests with ZrO_2 and ThO_2 . During the initial phase of the work, it was observed that ZrO_2 when heated to 1800°C for 1 hr in contact with Cm_2O_3 luminesced with a strong red glow throughout the 1/16-in.-thick pellet on which a curium oxide pellet had been placed. No reaction boundary layer was visible between the Cm_2O_3 and the ZrO_2 , indicating that the Cm_2O_3 penetrated the ZrO_2 by solid solution and diffusion. To estimate the concentration of Cm_2O_3 on the bottom side of the ZrO_2 pellet fired in contact with Cm_2O_3 , a mixture of Cm_2O_3 and ZrO_2 was ground, pressed, and fired to promote solution. This mixture which contained 12 wt % Cm_2O_3 glowed dull red due to luminescence. From the relative intensity of the luminescence, it was estimated that the bottom side of the ZrO_2 pellet contained more than 12 wt % Cm_2O_3 . By using these data, the diffusion coefficient (D) of curium in ZrO_2 (80% of theoretical density) was estimated to be $>1.1 \times 10^{-6} \text{ cm}^2/\text{sec}$ at 1800°C . In a similar experiment at 1400°C , curium penetrated a 1/16-in.-thick ZrO_2 pellet to 12 wt % concentration on the bottom side in 72 hr, indicating a diffusion coefficient of $>1.5 \times 10^{-8} \text{ cm}^2/\text{sec}$. Similar experiments with ThO_2 (96% of theoretical density) gave almost identical results; D was $>2.7 \times 10^{-8} \text{ cm}^2/\text{sec}$ and $>3.9 \times 10^{-6} \text{ cm}^2/\text{sec}$ at 1400°C and 1800°C , respectively.

The formula used in calculating the diffusion coefficient from the experimental observations is

$$c' = (c/2)[1 + \text{erf}(x/2\sqrt{Dt})] \quad (\text{ref. 2})$$

where

c = initial concentration of curium = 1.0

c' = concentration of curium at a particular point in the medium (ZrO_2 , ThO_2) into which curium diffuses and at a particular time

D = diffusion coefficient of curium in the designated oxide (ZrO_2 or ThO_2) (cm^2/sec)

t = time (seconds)

x = distance (cm)

erf = error function

This formula is based on the assumption of infinite dimensions of the diffusion couple, and its application to our specific case provides an approximation of the diffusion coefficient that is near enough correct to permit qualitative evaluation of the results.

Values for the diffusion coefficients of curium in ZrO_2 and ThO_2 at 900 and 1100°C were calculated by extrapolation of the values obtained at 1400 and 1800°C . By substituting these diffusion coefficient values in the above formula, the times required to obtain 1 mole % and 5 mole % concentrations of curium at a penetration depth of 1 mm into the ZrO_2 or ThO_2 wall were calculated and are shown in Table 2. The time for a

given curium concentration to occur within the diffusion medium is approximately proportional to the square of the depth within the medium.

Table 2. Time for 1.0 and 5.0 mole % Curium Concentration at 1-mm Depth in ThO_2 and ZrO_2

Temp (°C)	<u>Diffusion Coefficient (cm²/sec)</u>		Time in ThO ₂	Time in ZrO ₂
	²⁴⁴ Cm in ThO ₂	²⁴⁴ Cm in ZrO ₂		
<u>1.0 mole % Curium Concentration</u>				
1800	3.9 × 10 ⁻⁶	1.1 × 10 ⁻⁶	4 min	14 min
1400	2.7 × 10 ⁻⁸	1.5 × 10 ⁻⁸	10 hr	18 hr
1100	6.7 × 10 ⁻¹¹	8.4 × 10 ⁻¹¹	5.5 mo	4.4 mo
900	2.5 × 10 ⁻¹³	7 × 10 ⁻¹³	119 yr	43 yr
<u>5.0 mole % Curium Concentration</u>				
1800	3.9 × 10 ⁻⁶	1.1 × 10 ⁻⁶	7 min	27 min
1400	2.7 × 10 ⁻⁸	1.5 × 10 ⁻⁸	18 hr	33 hr
1100	6.7 × 10 ⁻¹¹	8.4 × 10 ⁻¹¹	10 mo	8 mo
900	2.5 × 10 ⁻¹³	7 × 10 ⁻¹³	219 yr	79 yr

The formation of solid solutions of ZrO_2 and ThO_2 with Cm_2O_3 had previously been predicted from phase diagrams of lanthanide oxides with ThO_2 and ZrO_2 . However, no phases having melting points of less than 1800°C were expected, and none were found. The diffusion coefficients are much greater than expected from data on other oxide systems. For example, the diffusion coefficient for curium in ZrO_2 , as shown in Fig. 3, is four orders of magnitude greater than that of zirconium ion in ZrO_2 . All diffusion coefficients have, however, almost identical activation energies (Q) of ~ 90 kcal/mole (see Fig. 3).

Fig. 3. Diffusion Coefficients.

Source Fabrication Facility Preparation

Decontamination of cell 5 and repair of its in-cell services were completed. A new back door seal-plate was installed. After the final encapsulation of ^{244}Cm residues for storage is completed, cell 5 will be checked for inert atmosphere control and equipment for ^{244}Cm compatibility sample welding will be installed. All equipment for pelletization, storage, and sample loading for the compatibility samples is on hand except for some miscellaneous tools. Cells 3 and 4 have been repaired to provide the proper inert atmosphere for encapsulation of the compatibility samples. The cell 3 inert atmosphere system has been checked out, but cell 4 has a small in-leak which has not yet been located.

Vapor Transport of $^{244}\text{Cm}_2\text{O}_3$

Mass spectra have been obtained with the mass spectrometer unit. A calibrated gas mixture has been prepared and used to tune the system. We are presently testing the unit with Nd_2O_3 to determine the lowest temperature at which NdO or Nd is first observed. Neodymium oxide is being used as a stand-in for $^{244}\text{Cm}_2\text{O}_3$ because it has been previously studied and probably has a vapor pressure similar to Cm_2O_3 . The NdO and Nd peaks have been observed at 1650°C .

It has been found that the lower the energy of the ionizing electrons the larger the neodymium oxide signal above the corresponding background at a particular mass point. Also the lower the energy of the ionizing electrons the less is the dissociation of the NdO due to the electron bombardment. It has been found that for this system an electron energy of 12 to 15 eV is the optimum level.

A shutter was installed to increase the signal-to-background ratio, and measurements were taken while measuring the intensity of the 158 mass peak (NdO). The electronics are now being checked to eliminate as much noise as possible and to smooth the signal obtained from the 158 mass peak. For the apparatus as presently constituted, the temperature at which the signal strength from the NdO peak (mass 158) is comparable to the variation in the noise level of the background peak at mass 158 is 1500°C . An additional increase in sensitivity will be obtained by the installation of a mechanical chopper in the spectrometer beam flight path. Delivery of the chopper mechanism has been delayed by the vendor until mid-January. The increased signal-to-background ratio is very important in measurements in the 1400°C range where the vapor specie beam strength is very small in relation to background noise.

Two electronic filters were added to the system which improved its performance. Measurement of the NdO mass peaks by the Knudsen method and extrapolation of the data using reported vapor pressures of Nd_2O_3 and Cm_2O_3 now indicate that the CmO mass peak can be observed at a specimen temperature of 1275°C . Using the Langmuir method with a specimen diameter of $3/8$ in., it is similarly estimated that the CmO mass peaks will be observed at specimen temperatures as low as 1100°C .

Helium Release from $^{244}\text{Cm}_2\text{O}_3$

Apparatus is being designed for the measurement of helium release rates from $^{244}\text{Cm}_2\text{O}_3$ pellets at temperatures below 1000°C . The steady-state helium release from $^{244}\text{Cm}_2\text{O}_3$ has been measured previously for the temperature range 900 – 1800°C . Extension of the data down to 500°C will provide important new information on fuel form behavior in power conversion devices designed for lower temperature operation for long periods of time. The apparatus consists of a custom-designed, resistance-heated furnace for inclusion in an existing alpha glove box facility. The furnace will have three heated positions which are independently temperature controllable for 500 , 650 , and 800°C operation. Each specimen will be contained in a molybdenum furnace tube connected to a vacuum manifold which will permit independent measurements of the helium release rate. At the conclusion of the heat-soak period, the molybdenum furnace tubes will be transferred to a high-temperature furnace for outgassing to 1800°C to determine the residual stored helium content.

Impact Testing of Heat Source Materials

The πMC particle size analyzer has been received and calibrated using a National Bureau of Standards standard slide. After calibration, a test run was made using the 5 - to $150\text{-}\mu\text{m}$ portion of a gadolinium oxide sphere that had been impacted cold at 234 ft/sec . Half of this sample has been sent to the Millipore Corporation for size analysis, and the other half was retained at ORNL. Our results, shown in Table 3, will be compared with the results from Millipore.

Table 3. Results of Particle Size Analysis

Size (μm)	Number of Particles (%)	Weight % in Size Range
>5	100	4.7×10^{-2}
>10	79.2	2.7×10^{-1}
>15	52.8	3.2×10^{-1}
>20	41.6	1.4×10^{-1}
>25	39.3	5.8
>50	18.5	30.1
>100	5.1	47.3
>150	0.56	16.0
>200	0	

With proper sample preparation, the instrument is capable of giving size distributions down to $0.1\text{ }\mu\text{m}$. The $<5\text{-}\mu\text{m}$ fraction of the Gd_2O_3 sample is presently being prepared for size analysis.

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