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ORNL ISOTOPIC POWER FUELS
QUARTERLY REPORT
FOR PERIOD ENDING JUNE 30, 1970
Eugene Lamb

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ISOTOPES DEVELOPMENT CENTER

ORNL ISOTOPIC POWER FUELS QUARTERLY REPORT
FOR PERIOD ENDING JUNE 30, 1970

Eugene Lamb

Isotopes Division

SEPTEMBER 1970

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
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for the
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SUMMARY

The Curium Source Test continued to operate at a temperature of approximately 1300°C on the fuel capsule and at a vacuum of approximately 7×10^{-3} torr in the Primary Vacuum Chamber. The pressure and temperature of the helium reservoir were recorded for 67 days following assembly of the Curium Source to determine if the helium was being released quantitatively as the curium fuel decayed.

Metallographic examination of two sets of $^{244}\text{Cm}_2\text{O}_3$ compatibility couples exposed for 10,000 hr at 1650 and 1850°C is complete. The effect of Gd_2O_3 on the phase change temperature of Cm_2O_3 is being studied. An adiabatic calorimeter is being assembled to measure the heat capacity of Cm_2O_3 .

Both nonradioactive and radioactive SrTiO_3 compatibility couples are being exposed at temperatures of 900 and 1100°C for various times. The thermal conductivity of a hot-pressed 2.48-g $^{90}\text{Sr}_2\text{TiO}_4$ pellet was measured.

High-pressure tests and temperature aging tests have been made on thin-walled SNAP-23 capsules and on heavy-walled threaded cap SNAP-23 capsules.

INTRODUCTION

The development of fuel forms with optimum design for use at temperatures up to 2000°C involves obtaining the characteristics of isotopic power and heat sources for anticipated applications in aerospace, terrestrial, and marine environments. The physical and chemical properties of the compound and source form, such as thermal conductivity, density, gas retention, and melting point, must be determined. Compatibility of the fuel form with container materials must be established to ensure adequate containment during the intended lifetime of the mission.

MATERIALS TECHNOLOGY AND DEVELOPMENT

Curium Fuel FormsFueled Curium Source Test

The Curium Source Test continued to operate at a temperature of approximately 1300°C on the fuel capsule and at a vacuum of approximately 7×10^{-3} torr in the Primary Vacuum Chamber.

The helium reservoir of the fueled Curium Source is equipped with a pressure monitoring instrument so that the release of helium from the curium fuel into the reservoir can be observed. The pressure and temperature of the helium reservoir were recorded for 67 days following assembly of the Curium Source. These data are shown in Fig. 1. The initial pressure in the reservoir at time zero was due to argon. The helium release rate calculated from curium decay gives a pressure increase in the reservoir of 0.72 mm Hg per day assuming an average reservoir temperature of 100°F. Pressure data for the first 11 days, when the average reservoir temperature was approximately 100°F, give a pressure increase of 0.75 mm Hg per day (see Fig. 2). At this point the cooling water to the baseplate was valved off causing the pressure in the reservoir to increase rapidly due to the increase in reservoir temperature. After 5 days of operation with the cooling water off, the pressure had increased to 1,119 mm Hg, and the reservoir temperature had stabilized at approximately 430°F. The pressure calculated from the ideal gas law and from the helium released in 5 days for the temperature change from 100°F to 430°F was 1,100 mm Hg (see point 1 in Fig. 1). Pressure data during the 5 days could not be analyzed to calculate a helium pressure increase per day due to the thermal instability of the reservoir. However, the pressure predicted by the ideal gas law is in close agreement with the actual recorded pressure.

Fig. 1. Curium Source Test History.

Fig. 2. Rate of Pressure Rise in Reservoir at 100°F.

The cooling water to the baseplate was turned on and after 3 days the pressure in the reservoir had dropped to 504 mm Hg. The average reservoir temperature was about 90°F. The gas law calculation and helium release for 3 days predict a reservoir pressure of 706 mm Hg (see point 2, Fig. 1). A discrepancy of 202 mm of Hg in the predicted pressure and the recorded pressure in the reservoir system was observed. At this point the cooling water was valved off and the 5 cartridge heaters were turned on. After 8 days the pressure in the reservoir was 756 mm Hg and the reservoir temperature was 575°F. Another discrepancy of 187 mm Hg was noted during this transient period (see point 3, Fig. 1). For the next 17 days the system was operated with the baseplate heaters on. The calculated helium release rate due to alpha decay would give a rate of pressure increase of 1.364 mm Hg per day at a temperature of 600°F compared with the observed rate of pressure increase of 1.31 mm Hg per day during the 17-day period (see Fig. 3).

Fig. 3. Rate of Pressure Rise in Reservoir at 600°F.

After the experiment had been in progress for 44 days, the baseplate heaters were turned off, and the reservoir was allowed to come to an equilibrium temperature of 430°F. The pressure of the reservoir dropped to 711 mm Hg, but the gas law predicted the pressure would drop to 659 mm Hg. This calculation shows a difference in pressure of 52 mm Hg, but the recorded pressure is higher than the calculated pressure (see point 4, Fig. 1). The pressure recorded on the 55th day indicated that the pressure did drop to a point predicted by the gas law and helium release. The extrapolation of the line at point 4 in Fig. 1 appears to fall very close to the pressure curve for the last 12 days of the period of observation. The rate of pressure increase during the last 12 days of the observation period was calculated to be 1.20 mm Hg per day. The theoretical rate of pressure increase at an average reservoir temperature of 440°F was 1.15 mm Hg per day (see Fig. 4).

Fig. 4. Rate of Pressure Rise in Reservoir at +430°F.

An analysis of the data shown in Figs. 1, 2, 3, and 4 led to the following tentative conclusions:

1. Helium was released quantitatively from the curium fuel and was accumulated quantitatively in the helium reservoir during the first 16 days of the experiment (see Fig. 2 and point 1 in Fig. 1).
2. A leak from the helium reservoir into the Primary Vacuum Chamber occurred between the 16th and 27th day.
3. Helium was released quantitatively from the curium fuel and was accumulated quantitatively in the helium reservoir during the time from the 27th to the 67th day (see Figs. 3 and 4). During the time from the 44th to the 54th day a temporary malfunction of the pressure-indicating instrumentation resulted in a high pressure indication (see point 4 in Fig. 1).

Subsequent to the operations described above, experiments were conducted to establish the cause of the above observations. These subsequent experiments established that:

1. The pressure in the helium reservoir was adequately recorded by the instrumentation (except for point 4 in Fig. 1).
2. The helium produced by the decay of the curium fuel was essentially all released to its helium reservoir.
3. An intermittent and variable leak exists from the helium reservoir into the Primary Vacuum Chamber.

Curium Fuel Development and Properties

Compatibility of $^{244}\text{Cm}_2\text{O}_3$. A summary of the status of the $^{244}\text{Cm}_2\text{O}_3$ compatibility test currently in progress is shown in Table 1.

Table 1. Current Status of $^{244}\text{Cm}_2\text{O}_3$ Compatibility Couples^a

Sample Series No.	Inner Capsule Material ^b	Exposure Conditions		Status
		Temp (°C)	Time (hr)	
IB-4	Refractory Metals A	1850	10,000	Metallography complete; preparation for electron microprobe analysis in progress
IB-3	Refractory Metals A	1850	1,000	Transferred to HRLEL
IA-2 ^a	Refractory Metals A	1650	10,000	Metallography complete; preparation for electron microprobe analysis in progress
IB-1	Refractory Metals A	1650	1,000	Sectioning of capsules complete; preparation for metallography in progress
IB-2	Refractory Metals A	1650	10,000	Exposure at temperature to end February 17, 1971
IB-6	Refractory Metals B	1250	1,000	Sectioning of capsules complete; preparation for metallography in progress
IB-8	Refractory Metals B	1250	10,000	Exposure at temperature to end January 24, 1971
IB-7	Superalloys	750	10,000	Exposure at temperature to end February 21, 1971

^aThe source of $^{244}\text{Cm}_2\text{O}_3$ is SRL except for Sample Series No. IA-2 which is from ORNL.

^bRefractory Metals A: Mo, T-111, Ta, Ta-10% W, W, W-26% Re, and TZM.

Refractory Metals B: Mo, Nb, Nb-1% Zr, Ta, Ta-10% W, V, and Zr.

Superalloys: Co-10% Cr, Ni-20% Cr, Ni-10% Cr, Hastelloy C, Haynes 25, Hastelloy N, and Ni.

Helium Release in $^{244}\text{Cm}_2\text{O}_3$. The results of steady-state helium release determinations at temperatures above 1610°C with 100% Cm_2O_3 are presented in Fig. 6. The activation energy for helium release changes very near the temperature of transformation from B-type monoclinic structure to

Fig. 5. Cm_2O_3 Apparent Diffusion Coefficient Versus Temperature.

A-type hexagonal structure. This effect is probably due principally to the change in the crystal structure at phase change, but a minor cause may be a decrease in the size of discrete crystallites.

Effect of Gd_2O_3 on Phase Change Temperature of Cm_2O_3 . The effect of the addition of various amounts of Gd_2O_3 in increasing the temperature of the B (monoclinic) to A (hexagonal) transition in Cm_2O_3 is being studied.¹ The temperature for the phase transition for the composition 54 mole % Gd_2O_3 -46 mole % $^{244}Cm_2O_3$ was $1860^{\circ}C$, which is $90^{\circ}C$ lower than the temperature predicted by extrapolation of data points shown in the previous quarterly report.¹ A sample of 70 mole % Gd_2O_3 -30 mole % $^{244}Cm_2O_3$ was run in an attempt to obtain an additional point that would permit better definition of the slope of the curve representing the data. This sample exhibited no phase change up to $2050^{\circ}C$ using the change in helium release rate as the indicator of occurrence of phase change. The data resulting from this series of experiments are plotted in Fig. 6. This phase of the work is complete except for confirmation of the data by X-ray diffraction which will be done when a high temperature stage is obtained for the existing diffractometer.

Fig. 6. Temperature of Phase Change (B-type to A-type) of $^{244}\text{Cm}_2\text{O}_3\text{-Gd}_2\text{O}_3$ Mixtures.

Volatile Transfer of Cm_2O_3 in a Helium Atmosphere. Measurements were made of the rate of volatilization and diffusion of curium from solid Cm_2O_3 in helium gas at 1900, 2000, and 2100°C. The observed rates, which were reported in the last quarterly report,¹ have been correlated by Eq. (1) so that the value of $(D)(C)$ can be calculated for temperatures other than those at which the data were taken:

$$\log_{10} \left[\frac{(D)(C)}{T^{1/2}} \times 10^{10} \right] = 14.324 - \frac{29,050}{T} \quad (1)$$

where

D = diffusion coefficient, cm^2/sec

C = concentration of Cm_2O_3 , g/cm^3

T = temperature, $^\circ\text{K}$.

This equation was derived assuming that the vapor pressure varies with temperature according to the equation of Smith and Peterson.² The diffusion coefficient was assumed to vary as the 1.5 power of the absolute temperature. The data were reported previously as grams of curium/cm.sec while Eq. (1) is in grams of $\text{Cm}_2\text{O}_3/\text{cm}.\text{sec}$.

According to Smith and Peterson, Cm_2O_3 vaporizes by one or both of the following reactions:

Oxygen is largely in the monatomic form at the temperatures and pressures of this work.³ In either of the above reactions, the equilibrium partial pressure of the molecular species of curium is dependent on the partial pressure of the oxygen. Consequently, the diffusion of oxygen away from the Cm_2O_3 surface will increase the partial pressure and the gross rate of diffusion of curium. The effective diffusion coefficient for Cm_2O_3 transfer will depend on the diffusion coefficients of all molecular species present. With the present data, specific diffusion coefficients cannot be determined. Only the product, (D)(C), could be determined; however, transfer rates can be calculated when saturation vapor concentration is present at one end of the path and the concentration at the other end of the path is negligible by comparison.

Some separation of trace impurities was observed in the volatility tests. Iron, potassium, magnesium, manganese, and nickel were present in substantially higher concentrations in the sublimed material than in the original Cm_2O_3 . On the other hand, tantalum and zirconium did not sublime to any appreciable degree. The americium concentration was 1.6 fold higher in the sublimed material than in the original material. It is probable that all of these materials were present as the oxides.

Calculations based on these latest data were made of the diffusion rate through a 0.1-in.-diam capillary. The calculations assumed 1 atm of helium pressure and a temperature of 1800°C . This is the temperature of the coolest surface over which the Cm_2O_3 will pass before reaching the capillary entrance. Two higher temperatures are included to indicate the performance of the source at higher temperatures. The amount of Cm_2O_3 required to plug the capillary was estimated to be 0.14 g. The plug was assumed to be 0.1 in. in diameter and 0.1 in. thick. The values of the product, (D)(C), were calculated by Eq. (1). The data are given in Table 2. The figures apply only to Cm_2O_3 moving by diffusion. They do not apply to foreign materials which may be present or to Cm_2O_3 transported by bulk movement of helium through the capillary. This bulk flow occurs during changes of temperature of the helium tank. The effect of flow caused by helium generation is negligible.

Table 2. Transport of Cm_2O_3 Through Capillary

Temperature (°C)	Rate of Transport (g/day)	Days to Plug 0.1-in. Capillary
1800	2.6×10^{-6}	540,000
1900	1.3×10^{-5}	9,500
2000	5.5×10^{-3}	390

Autoradiographs of the tungsten tubes in which the sublimed Cm_2O_3 condensed indicated that the radioactive material in two of the three tubes was in the form of four distinct bands. (The autoradiographic technique provided a better definition than the previously reported gamma scanning technique,¹ which indicated the presence of two to four bands of radioactivity.) Three of the four bands may consist of fission products or other radioactive impurities. The absorption of gamma rays by the tungsten tubes interfered seriously with identification. Experiments are planned in which the Cm_2O_3 vapor diffuses along a path of annular cross section. There will be a longitudinal temperature gradient in the region in which condensation occurs. The inner surface of the annulus will be slightly cooler than the outer surface so that most of the material will condense on the inner surface. This surface (a tungsten rod) will be removed and examined visually, and gamma scans will be used to identify radioactive materials. The nonradioactive materials which could cause capillary plugging can also be identified. A test of the apparatus using Al_2O_3 and iridium indicated that the materials condensed on the inner surface as predicted.

The temperature gradient along the annular passage will be measured by means of melting point standards. These standards will be in the form of fine wires pressed into grooves in the inner surface of the annular passage.

Viscosity and Surface Tension of $^{244}\text{Cm}_2\text{O}_3$. A detailed specification for the high-temperature vacuum furnace system for the viscosity surface tension apparatus has been drafted covering the general capability, dimensional limitation, and special features.

Experiments have been conducted at room temperature to investigate the technical feasibility of using a Cahn electrobalance to measure viscosity. Both glycerine and water were used in the experiment. The first results indicate that the distance traveled by the bob is too short for the desired accuracy. The experimental accuracy was improved by use of a cathetometer.

Heat Capacity of $^{244}\text{Cm}_2\text{O}_3$. The assembly of the adiabatic calorimeter for the measurement of the heat capacity of $^{244}\text{Cm}_2\text{O}_3$ is complete. A procedure for the fabrication of $^{244}\text{Cm}_2\text{O}_3$ samples for the calorimeter with the thermocouple fabricated integral with the sample has been developed. The thermocouple also performs the function of suspending the sample within the calorimeter.

CVD Tungsten Capsule Furnace Test.

This experiment was begun March 24, 1970. After 39 days of the planned 90-day test, the pressure was 115 psig and the temperature was 1650°C; the furnace pressure was 7.3×10^{-7} torr. Sixty-five days after the start of the experiment, the pressure was 180 psig and the temperature was being held constant at 1650°C. The furnace pressure had dropped to 5.8×10^{-7} torr. The tungsten capsule ruptured on the 88th day of the test. The capsule pressure at the time of rupture was 245 psig. The length of the capsule had increased from 2.2028 to 2.2054 in., and the diameter at the capsule's midsection had increased from 0.7935 to 0.9163 in. (see Fig. 7).

Fig. 7. CVD Tungsten Capsule Involved in 90-day Test.

Strontium Fuel Forms

Compatibility of $^{90}\text{SrTiO}_3$

The exposure time as of June 29, 1970, for two $^{90}\text{SrTiO}_3$ compatibility couples at 1100°C is approximately 3000 hr, while that for one couple at 900°C is approximately 2940 hr.

The encapsulation of four nonradioactive SrTiO_3 compatibility couples (container material: Hastelloy C) was completed. The data from these couples which were assembled from SrTiO_3 prepared by the K_2CO_3 precipitation process will be compared with the data previously acquired on the compatibility of SrTiO_3 prepared by the $(\text{NH}_4)_2\text{CO}_3$ process to determine the influence of the SrTiO_3 preparation method on the compatibility of SrTiO_3 with Hastelloy C. These four couples are now being exposed in a muffle furnace at 900°C for 10,000 hr and at 1100°C for 1,000, 5,000, and 10,000 hr.

Compatibility of SrO

The effect of varying the amount of silicon in Hastelloy C on its compatibility with SrO prepared by both the $(\text{NH}_4)_2\text{CO}_3$ and K_2CO_3 precipitation processes was studied by exposure of nine compatibility couples (listed in Table 3) at 1100°C for 1000 hr.

The metallography was completed for the nine nonradioactive SrO -Hastelloy C couples. Examination of the photomicrographs has confirmed that the extent of attack on Hastelloy C by the SrO pellets prepared by the K_2CO_3 process is considerably greater than those from the $(\text{NH}_4)_2\text{CO}_3$ process.

Table 3. Study of Compatibility of Hastelloy of Various Silicon Contents With Nonradioactive SrO Fuel Material^a

Precipitation Process	Temperature (°C)	Time (hr)	Container Material
(NH ₄) ₂ CO ₃	1100	1000	Hastelloy containing 0, 0.5, 1.0, and 2.0% Si and Hastelloy C-276
K ₂ CO ₃	1100	1000	Hastelloy containing 0, 0.5, 1.0, and 2.0% Si

^a A muffle furnace exposed to air was used for the study. The double-encapsulated compatibility couple was contained in a thick-walled (~1/4 in.) Inconel capsule before exposure.

The attack was most severe for the Hastelloy C alloy containing 0.5 wt % silicon, but it became less extensive for alloys with the silicon content either higher or lower than this value.

Capsules of Hastelloy C and Hastelloy C-276 are being prepared for testing with ⁹⁰SrO that will be made by both precipitation processes. These tests are scheduled to operate for greater than 1,000 and 10,000 hr at 750 and 1100°C (see Table 4).

Table 4. Study of the Compatibility of Hastelloy of Various Silicon Contents with ⁹⁰SrO Fuel Material

Type of Compound	Precipitation Process	Temperature (°C)	Time (hr)	Container Material
⁹⁰ SrO	K ₂ CO ₃	750	>1,000	Hastelloy C-276
⁹⁰ SrO	K ₂ CO ₃	750	>10,000	Hastelloy C-276
⁹⁰ SrO	K ₂ CO ₃	1100	>1,000	Hastelloy C-276
⁹⁰ SrO	K ₂ CO ₃	1100	>10,000	Hastelloy C-276
⁹⁰ SrO	K ₂ CO ₃	1100	>1,000	Hastelloy C
⁹⁰ SrO	K ₂ CO ₃	1100	>10,000	Hastelloy C
⁹⁰ SrO	(NH ₄) ₂ CO ₃	750	>1,000	Hastelloy C-276
⁹⁰ SrO	(NH ₄) ₂ CO ₃	750	>10,000	Hastelloy C-276
⁹⁰ SrO	(NH ₄) ₂ CO ₃	1100	>1,000	Hastelloy C-276
⁹⁰ SrO	(NH ₄) ₂ CO ₃	1100	>10,000	Hastelloy C-276
⁹⁰ SrO	(NH ₄) ₂ CO ₃	1100	>1,000	Hastelloy C
⁹⁰ SrO	(NH ₄) ₂ CO ₃	1100	>10,000	Hastelloy C

Thermal Conductivity of $^{90}\text{Sr}_2\text{TiO}_4$

The thermal conductivity of a 24.8-g pellet of $^{90}\text{Sr}_2\text{TiO}_4$ that had been hot pressed at 1300°C for 30 min to a density of 4.26 g/cm³ at the Fission Products Development Laboratory was measured. The heat generation rate of the pellet measured in a boiling water calorimeter was 6.034 W. The chemical analysis of the pellet is shown in Table 5. The pellet contained 95% of the stoichiometric amount of TiO_2 needed for the composition, R_2TiO_4 , where R is alkaline earth element.

The thermal conductivity of the pellet was measured in an absolute thermal conductivity apparatus using the heat generated by the beta decay of ^{90}Sr and ^{90}Y as a known heat source for the measurements. The method and apparatus have been described previously.^{4,5} The thermal conductivity values at various temperatures are shown in Table 6. The data from Table 6 have been fitted to an equation by the least-mean-squares method. The equation is:

$$1/k = 56.705 + 0.01255T \quad (2)$$

where T is in °K and k is in W/cm.°C.

Table 5. Chemical Composition of $^{90}\text{Sr}_2\text{TiO}_4$

Compound	Amount (wt %)
SrO	65.5
CaO	6.2
TiO_2	27.8
ZrO_2	0.5
MgO	nil
BaO	nil

Table 6. Thermal Conductivity of Sr_2TiO_4

Temperature (°C)	k (W/cm.°C)
440	0.0151
519	0.0151
522	0.0150
680	0.0143
746	0.0148
866	0.0139

The 95% confidence band was calculated from Table 6 and Eq. (2). The plot of Eq. (2) [thermal resistivity (1/k) versus temperature] and the 95% confidence band are shown in Fig. 8. The thermal conductivity of $^{90}\text{Sr}_2\text{TiO}_4$ from Eq. (2) and the thermal conductivity of $^{90}\text{SrTiO}_3$ (ref. 5) are shown in Fig. 9 as a function of temperature. The slope of the thermal conductivity with temperature for $^{90}\text{Sr}_2\text{TiO}_4$ is much less than the slope for $^{90}\text{SrTiO}_3$. The same lower slope was observed with inactive Sr_2TiO_4 as compared with inactive SrTiO_3 (refs. 6 and 7).

Thermal Conductivity of $^{90}\text{SrSiO}_3$ Source Material

The thermal conductivity of $^{90}\text{SrSiO}_3$ material used in the preparation of beta sources was determined. The $^{90}\text{SrSiO}_3$ source material contains approximately 12 mole % additives to alter the properties of the $^{90}\text{SrSiO}_3$. The additives, Li_2O , Al_2O_3 , K_2O , and V_2O_5 , are present in concentrations of approximately 1 to 5 mole %.⁸

Fig. 8. Correlation of Thermal Conductivity Data for Sr_2TiO_4 .

Fig. 9. Comparison of Thermal Conductivity of $^{90}\text{SrTiO}_3$ and $^{90}\text{Sr}_2\text{TiO}_4$.

A 1-in.-diam, 24.74-g pellet was prepared by hot pressing 100 mesh powder at 825°C and 4000 psi to a density of 3.02 g/cm³. The thermal power of the pellet measured with a boiling water calorimeter was 4.68 W.

The thermal conductivity of the pellet was measured in an absolute thermal conductivity apparatus using the heat generated by the beta decay of ⁹⁰Sr and ⁹⁰Y as a known heat source for the measurements. The method and apparatus have been described previously.^{4,5} The thermal conductivity values at various temperatures are shown in Table 7. The data from Table 7 have been fitted by the least-mean-squares method to the equation:

$$1/k = 0.07075T - 0.772 \quad (3)$$

where T is in °K and k is in W/cm.°C.

The 95% confidence band was calculated from Table 7 and Eq. (3). The plot of Eq. (3) [thermal resistivity (1/k) versus temperature] and the 95% confidence band are shown in Fig. 10. The thermal conductivity of ⁹⁰SrSiO₃ from Eq. (3) and thermal conductivity of ⁹⁰SrTiO₃ (ref. 4) are shown in Fig. 11 as a function of temperature. The slope of the thermal conductivity with temperature for ⁹⁰SrSiO₃ is greater than the slope for ⁹⁰SrTiO₃. The thermal conductivity of SrSiO₃ is about one-third that of SrTiO₃ over the temperature range 600-900°C. The thermal conductivity of ⁹⁰SrSiO₃ material at 700°K is 0.0205 W/cm.°C compared with the thermal conductivity of pyrex glass which is 0.0165 W/cm.°C at 700°K.

Fig. 10. Correlation of Thermal Conductivity Data for ⁹⁰SrSiO₃.

Table 7. Thermal Conductivity of a $^{90}\text{SrSiO}_3$ Specimen

Temperature ($^{\circ}\text{C}$)	Thermal Conductivity ($\text{W/cm}^{\circ}\text{C}$)
447	0.01883
570	0.01697
707	0.01447
785	0.01426
906	0.01174

Fig. 11. Comparison of Thermal Conductivity of $^{90}\text{SrSiO}_3$ with $^{90}\text{SrTiO}_3$.

Strontium Applications and Prototype Sources

SNAP-23 Program. High-pressure tests and temperature aging tests have been made on thin-walled SNAP-23 capsules and on heavy-walled threaded cap SNAP-23 capsules. A total of nine capsules have been tested. A chronological sequence of the tests made on these sources is given below:

1. A SNAP-23 liner containing a LAVA-A fuel simulant was subjected to high pressure tests of 9,200 psig and 16,100 psig. The source was leak tested before and after each test by the vacuum leak test method. No leaks were detected. After the first test, the liner had collapsed tightly around the LAVA-A fuel simulant. Three ridges were formed longitudinally along the source as the side wall collapsed. The side walls also pulled in at the top and bottom of the source. No distortion of the top and bottom of the capsule was observed. There was no noticeably visible change

in the source after the 16,100-psig test. The source was sent to Hittman Associates, Inc., for further examination.

2. A second SNAP-23 liner, also loaded with a LAVA-A fuel simulant, was placed in a furnace, initially at room temperature, and the temperature was programmed to increase to 815°C (1500°F). After approximately 2-1/2 hr and a furnace temperature of 400°C (752°F), the ends of the capsule were severely bulged with some distortion also observed in the cylindrical side walls. A small hole was drilled in one end to relieve gas pressure buildup in the source (water vapor was observed escaping from the hole by holding a cool glass over it and observing condensation on the glass). The capsule was returned to the furnace [furnace temperature 450°C (842°F)]. The capsule remained in the furnace for another 2-1/2 hr until the temperature reached 700°C (1292°F) at which time the top of the capsule blew off from overpressurization.
3. A third SNAP-23 liner with a LAVA-A fuel simulant was prepared for the furnace test by drilling a 1/8-in.-diam hole in the top to relieve gas pressure buildup and was placed in the furnace. The furnace was programmed to rise from 200°C (392°F) to 815°C (1500°F) in 4 hr. The capsule remained at this temperature for 108 hr. At the end of this time the furnace door was opened and the capsule pulled partially out of the furnace. While watching the capsule cool (by observing the color change of the metal capsule), the top of the capsule popped off and within a few minutes the bottom of the capsule also popped off. The welds were broken all the way around the capsule. After the capsule was completely cool, the capsule outer diameter was 0.040 in. greater than it was originally. The LAVA-A fuel simulant was wedged tightly into the cylinder, where it had been a loose fit before the test. A check with the manufacturer by Hittman Associates personnel revealed that the LAVA-A material could be expected to permanently "grow" 1% when heated to 1500°F (815°C).

Six capsules of three designs were received from Hittman Associates for testing. Two designs were thin-walled capsules made from the original SNAP-23 liners but with newly designed weld closures. One was a cupped design and the other a trepanned design. The third design was a heavy-walled capsule with screw-threaded caps that were seal welded. There were two capsules of each design sent to ORNL, three of which were to be pressure tested only and three of which were to be subjected to 815°C (1500°F) for 100 hr and then subjected to pressure tests. Five of the six capsules contained LAVA-A fuel simulants. The sixth capsule, one of the heavy-walled threaded end cap design, was empty and was subjected to the pressure test only.

4. Capsule No. C-1011-0002-3 (cupped end, thin wall) was subjected to pressure tests of 11,300 psig and 16,100 psig. No leaks were detected as a result of either of these tests. The capsule collapsed very similarly to the liner described previously with four

longitudinal ridges instead of three and the same flaring of the top and bottom because of the rigidity of the end caps.

5. Capsule No. C-1011-0002-15 (trepanned end, thin wall) was subjected to the same pressure tests as the capsule above with very nearly identical results.
6. Pressure tests on the heavy-walled screw thread capsule (No. C-1013C-0002-13), which contained no LAVA-A fuel simulant, were performed. The capsule was first subjected to a test at 11,300 psig. There were no leaks as a result of this test and no discernable deformation of the capsule; however, during the 16,100 psig test, the capsule ruptured. The rupture occurred at approximately 13,000 psig. The bottom, having no support from a fuel simulant, caved in, breaking the bottom of the trepan on the seal weld and pulling the threads out onto their edges which sheared. Had a fuel simulant been inside the capsule, it is likely that it would have passed the test.
7. Capsule No. C-1011-0002-14 (trepanned end, thin walled) was subjected to a temperature test at 815°C (1500°F) for 72 hr. The capsule was removed from the furnace when the furnace temperature was 200°C (392°F). A small crack in the heat-affected zone of the weld was observed when the source was removed from the furnace. Upon air cooling, the crack extended approximately half way around the capsule, across the weld for 3/4 in. in the bottom of the trepan, back across the weld, and down the entire length of the capsule. The capsule had a 1/8-in.-diam hole drilled in the top cap prior to the test to vent gaseous pressure buildup. There was no bowing of the caps as a result of the test.
8. Capsule No. C-1013C-0002-12 (heavy-walled, threaded cap) was subjected to a 100-hr test at 815°C (1500°F). The capsule had a 1/8-in.-diam hole in the top cap to vent gaseous pressure buildup, but it was bowed on the bottom cap when removed from the furnace. The vent hole was plugged and welded and the capsule was leak tested; no leaks were observed. The capsule was then subjected to a pressure test. At a pressure of 8400 psig the capsule was heard to crack. A leak test disclosed that the crack had occurred at the bottom corner of the seal weld trepan on the bulged end.
9. Capsule No. C-1011-0002-2 (cupped, thin wall) was subjected to a 100-hr temperature test at 815°C (1500°F). This capsule was cooled by programming the temperature down 100°C (212°F) at 8:00 each morning and again at 4:00 each evening. The temperature was programmed down to 300°C (572°F) on the evening of the third day. The next morning the end caps had cracked off.

A stress analysis was performed on the modified SNAP-23 capsule which was proposed earlier.⁹

A summary of stresses produced by an external pressure of 10,000 psi of the various components of the capsule is shown below:

1. Center of bottom cap
 - a. Outside 62,900 psi
 - b. Inside 42,900 psi
2. In bottom weld
 - a. $t_w = 0.110$ 19,700 psi
 - b. $t_w = 0.080$ 38,800 psi
3. Thickness of threaded cap
 - a. Required 0.996 in.
 - b. Actual 1.000 in.
4. Number of threads
 - a. Minimum required 2
 - b. Actual 4
5. Rotation of head due to pressure of 10,000 psi if unrestrained 0.0016 in.
6. Stresses in cylinder remote to ends
 - a. Circumferential 6.11×10^4 psi
 - b. Longitudinal 3.34×10^4 psi
 - c. Radial 0.566×10^4 psi

Materials Oxidation Studies

The eleventh and twelfth sets of air oxidation coupons of Hastelloy X, TD Nichrome, and Nichrome series were removed from the furnace on April 13 and May 12, 1970, respectively, after exposure to air at 1000°C for 11 and 12 months, respectively. The fourth set of soil oxidation coupons of Hastelloy X, TD Nichrome, and Nichrome was removed from the furnace on May 12, 1970, after exposure to soil at 1000°C for 12 months. Data for these coupons are given in Table 8.

Table 8. Oxidation of Hastelloy X (HX), TD Nichrome (NT), and Nichrome (N) Coupons

Coupon Number	Weight (g)			Percent
	Original	New	Loss	Loss
<u>Air Coupons</u>				
HX-16	3.7954	3.7588	0.0366	0.964
NT-15	3.7981	3.7798	0.0183	0.482
N-15	3.6380	3.6001	0.0379	1.042
HX-15	3.7901	3.7271	0.0630	1.662
NT-16	3.8697	3.8521	0.0176	0.455
N-16	3.5967	3.5467	0.0500	1.390
<u>Soil Coupons</u>				
HX-4	3.7953	3.7397	0.0556	1.465
NT-4	3.8165	3.7271	0.0894	2.342
N-4	3.6256	3.5860	0.0396	1.092

Cladding Materials Program*

Development of Improved Alloys

Seven ternary platinum-base alloys with the nominal compositions listed in Table 9 were prepared by arc melting. Platinum atoms form the ordered phases Pt_3Ti and Pt_3Hf with titanium and hafnium atoms, respectively. The critical temperature for ordering of Pt_3Ti is much higher ($T_c = 1950^\circ C$) than that for alloy S-19, Pt_3Cr , ($T_c = 1070^\circ C$). Therefore, adding titanium to alloy S-19 is expected to increase the critical temperature for ordering for alloys 1, 2, and 3 to 1230, 1360, and $1480^\circ C$, respectively.

Table 9. Experimental Platinum Alloys

Alloy	Composition (wt %)	Alloy State	Critical Temperature for Ordering ^a ($^\circ C$)	Rockwell A Hardness
1	Pt-7.4 Cr-0.6 Ti	Ordered $Pt_3(Cr, Ti)$	1230	52.5
2	Pt-6.1 Cr-1.8 Ti	Ordered $Pt_3(Cr, Ti)$	1360	57.5
3	Pt-4.8 Cr-3.0 Ti	Ordered $Pt_3(Cr, Ti)$	1480	64.3
4	Pt-6 W-0.5 Hf	Solid solution and Pt_3Hf	—	54
5	Pt-6 W-1 Hf	Solid solution and Pt_3Hf	—	—
6	Pt-5 Mo-0.5 Hf	Solid solution and Pt_3Hf	—	54
7	Pt-5 Mo-1 Hf	Solid solution and Pt_3Hf	—	55

^aCalculated values.

The kinetics of the transformation for disorder to order in the ordered alloys was examined by Debye-Scherrer X-ray diffraction. Two important results were obtained. First, the ordering reaction in these ternary alloys is fast, and the disordered state cannot be retained by oil quenching from $1520^\circ C$. In the quenched condition, the intensity of superlattice reflections indicates that the formation of long-range order is more pronounced in the alloy 3 than in the other two alloys. Secondly, the rate of grain growth, even at temperatures above the critical temperature such as $1520^\circ C$, decreases with increasing of titanium content in these alloys. These results can be explained on the basis of an increasing critical temperature for ordering with increasing titanium content in the alloys (see Table 9).

As also indicated in Table 9, the ordered alloys are hardened effectively by partially replacing chromium by up to 3 wt % titanium. Although these alloys, prepared in the 20-g button form, cannot be cold-rolled satisfactorily because of cracks that form intergranularly, their forgeability improved with the addition of titanium. In fact, alloy 3, to which 3 wt % titanium was added, was forged successfully at room temperature.

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We expect alloys 4 through 7, which contain 0.5 to 1 wt % hafnium, to be strengthened by both solid solution and particles of Pt_3Hf . We also expect the stable particle Pt_3Hf to inhibit grain growth during aging at high temperature. The fabricability of alloys 4 through 7 is excellent. The two alloys with 0.5% hafnium were cold-rolled to 0.030-in.-thick sheet. These results indicate that the ordered phase Pt_3Hf does not impair the forgeability of the platinum-base alloys. The alloy sheet will be used for metallurgical study and evaluation of mechanical properties.

The oxidation behavior of ordered $\text{Pt}_3(\text{Cr}_{0.84}\text{W}_{0.16})$ and disordered Pt-6% W-1% Hf alloys was studied at 1000°C in air, and the results are shown in Fig. 12 with the results for Pt_3Cr , which were reported previously.¹⁰ The net weight change of platinum-base alloys on oxidation is a sum of the weight loss due to the formation of volatile platinum oxide and the weight gain due to the formation of a refractory oxide. The average weight gain of $\text{Pt}_3(\text{Cr}_{0.84}\text{W}_{0.16})$ was $6.5 \times 10^{-4} \text{ mg/cm}^2\cdot\text{hr}$. Alloys Pt-6% W-1% Hf and Pt_3Cr lost weight at rates of 6.8 and $9 \times 10^{-4} \text{ mg/cm}^2\cdot\text{hr}$, respectively. In contrast to the other alloys, no apparent oxide layer was observed on the surface of alloy Pt-6% W-1% Hf after a 100-hr exposure in air. Since unalloyed platinum loses weight at a rate of $7 \times 10^{-4} \text{ mg/cm}^2\cdot\text{hr}$, we conclude that the alloying with a limited amount of refractory elements does not impair the excellent resistance of pure platinum to oxidation.

We designed an apparatus for tensile testing experimental alloys in vacuum at high temperatures. The main part is a quartz tube furnace heated by an induction unit. The vacuum in the system is maintained by a VacIon pump. This apparatus was constructed and installed and is nearly ready for operation.

Fig. 12. Weight Change of Platinum-Base Alloys in Air at 1000°C .

Back Extrusion of Refractory Metal Capsules

A simple and inexpensive method for fabricating refractory-metal capsule shapes is desired, especially for the less fabricable molybdenum alloys. The back extrusion technique offers these advantages and, in addition, (1) does not require expensive sheet as a starting material as does deep drawing, (2) produces a tough, worked structure, and (3) commits only a small amount of material to each operation (unlike the extrusion of tubes). The purpose of this task is to demonstrate the capabilities of this process.

This process involves the extrusion of a solid blank into a closed die and back over an advancing plunger. A cross section of the tooling and its placement in the existing container sleeve of our extrusion press are shown in Fig. 13. The advantages of this process are that capsules with large diameters and closed ends can be fabricated from a simple blank, that the geometry can be changed by relatively simple changes in the design of the die and plunger, and that a large amount of deformation is possible below the recrystallization temperature so that a wrought structure with good mechanical properties is produced.

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Fig. 13. Tooling for Back Extrusion of Refractory Metal Capsules.

A number of capsules of Nb-1% Zr, Mo, and Mo-0.5% Ti that are nominally 2.5 in. in outside diameter, 5 in. long, and 0.25 in. in wall thickness and have hemispherical ends were fabricated. These were back extruded at extrusion loads of 150 to 300 tons on starting blanks cut from extruded and rolled bar stock heated to 1200°C in a salt bath. Cross-sectional and end views of a molybdenum capsule are shown in Fig. 14.

More recently we have concentrated on evaluating the back extrusion process as an economical method for fabricating Ta-10% W and T-111 isotope capsules. Twelve starting blanks of each material were cut from extruded bar stock for back extrusion of capsule bodies and end caps. The blanks were annealed 1 hr at 1600°C and plasma spray coated with molybdenum.

Fig. 14. Cross Section and End View
of Molybdenum Capsule Fabricated by Back Extrusion.

This coating protects the back extrusion blanks from contamination during preheating in an argon atmosphere and subsequent fabrication in air. Metallographic and chemical analyses showed that the resulting interaction layer is less than 0.010 in. deep. This layer is easily removed by machining.

Three Ta-10% W capsule bodies and four T-111 end caps were back extruded at 1450°C with a maximum stem load of 500 tons. Under these conditions the capsule bodies back extruded to a length of 4 to 4-3/4 in. These parts will be re-extruded to produce the required 5-1/2-in.-long capsule bodies. The plunger design is being modified to reduce friction enabling the back extrusion of longer capsules in a single step.

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