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by

W. C. Mosley, P. K. Smith, and P. E. McBeath

Savannah River Laboratory
E. I. du Pont de Nemours and Co.
Aiken, South Carolina 29801

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Savannah River Laboratory
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Palladium- ^{252}Cf oxide cermet has been developed as a versatile, safe source form for ^{252}Cf with low contamination potential. Developmental wire containing 1.85 mg of ^{252}Cf has been sold in three orders to U. S. commercial encapsulators. Bare palladium- ^{252}Cf oxide cermet wire containing ^{252}Cf concentrations of 5, 50, or 500 $\mu\text{g}/\text{inch}$ is expected to be offered for general sale in the near future.

INTRODUCTION

^{252}Cf is being developed by the AEC as an intense neutron source [2.31×10^{12} n/(g sec)] to be available at a price of \$10/ μg unencapsulated for a variety of industrial, medical, and nuclear applications (1). In support of an AEC ^{252}Cf source loan program, the Savannah River Laboratory (SRL) has fabricated industrial sources with up to 10 mg of ^{252}Cf as purified oxide precipitated and calcined in preselected quantities in a Pt-10% Rh capsule enclosed in an outer capsule of Type 304L stainless steel or Zircaloy-2 (2).

Bulk quantities of ^{252}Cf are also available in product capsules in which oxide or oxy sulfate is confined between two platinum frits in a platinum alloy tube (3). Source fabricators extract the ^{252}Cf from product capsules with nitric acid for chemical subdivision and fabrication into sources of desired strengths. Because these wet chemical operations must be performed for every source made and the operations have relatively high potential for contamination, and, hence, require expensive containment

facilities, a source form was desired which provides self-containment of bulk ^{252}Cf and which could be quantitatively subdivided without wet chemistry.

Palladium- ^{252}Cf oxide cermet was developed as a versatile, safe source form for bulk ^{252}Cf (Fig. 1)(4). Quantitative subdivision and encapsulation of bulk cermet wire, rod, or sheet containing a uniform dispersion of ^{252}Cf can be performed mechanically with less contamination hazard than from chemical processing. This paper describes the materials selection, cermet fabrication processes, properties of cermet product, and wire production experience.

A total of 1.85 mg of ^{252}Cf from the 3.5 mg fabricated into wire to demonstrate the processes has been sold to domestic encapsulators. General production and sale of wire at 5, 50, and 500 μg $^{252}\text{Cf}/\text{inch}$ and pellets with <10 mg of ^{252}Cf is expected to begin in the near future (5). The specifications for commercial wire are listed in Table I.

A. Wire

B. Autoradiograph

C. Densitometer Analysis of Radiograph

D. Neutron Counting Analysis

Fig. 1 Pd-²⁵²Cf Oxide Cermet Wire

TABLE I

Proposed Specifications for
Pd-²⁵²Cf Oxide Cermet Wire

²⁵² Cf Concentration	Nominally 5, 50, or 500 µg/inch
Total ²⁵² Cf	Within 10% of order, assayed to ±3% for pricing
Thickness	0.040 ±0.002-inch-square cross section
Length	0.5 to 2.5 inches
Uniformity	1/8 inch segments within ±10% of average concentration
Metallurgical Condition	Annealed

Selection of Palladium Matrix

The concept of dispersing ²⁵²Cf in a noble metal was selected because the matrix should have resistance to oxidation and corrosion, a high melting temperature, and good ductility. Palladium was chosen specifically because its low cross section for thermal neutron capture of 8 barns and low capture gamma interference parameter (6) of 1.48 would minimize interference with activation analyses. Its solubility in oxidizing

acids also would facilitate ²⁵²Cf recovery.

Process Development and Demonstrations

Initial efforts at developing an improved ²⁵²Cf neutron source form were directed towards preparation of palladium-californium alloy. However, because of the reported high volatility of metallic californium (7) and the difficulties experienced in reducing stand-in samarium oxide and alloying it with palladium, the alloy process was temporarily abandoned in favor of developing a cermet form. However, the formation of ductile palladium-californium alloy has been demonstrated subsequently by melting Pd-²⁵²Cf oxide cermet in vitreous carbon crucibles to effect simultaneous carbothermic reduction of oxide and alloying with palladium with quantitative assimilation of ²⁵²Cf (8) into the Pd alloy button. Wires made in this manner with samarium stand-in indicate the potential for accommodating over 10 times the ²⁵²Cf source strength as for wires made from cermet as described below. However, such high source strengths are not required for the present ²⁵²Cf applications, and SRL has no plans to produce the alloy for the sales program.

Several cermet preparation methods were investigated. They include evaporation of ²⁵²Cf nitrate onto palladium powder and conversion to ²⁵²Cf oxide, chemical plating of palladium on ²⁵²Cf oxalate particles and conversion to ²⁵²Cf oxide, and freeze drying of californium-palladium nitrate solution and conversion to ²⁵²Cf oxide and palladium.

Because of simplicity and high yields in remote operation, the chemical plating process was chosen for routine production of Pd-²⁵²Cf oxide cermet. The chemical plating process is similar to a process used by Fuschillo and Gimpl (9) to prepare dispersion-hardened alloys of platinum and gold. A flow-sheet for producing Pd-²⁵²Cf oxide cermet wire is given in Figure 2. Samarium stand-in and ²⁵²Cf tracer were used for development prior to demonstration of the process in shielded facilities using milligram quantities of ²⁵²Cf. Table II

describes the Pd-²⁵²Cf oxide cermet wires fabricated during the in-cell demonstrations.

Fig. 2 Chemical Plating Process to Fabricate Pd-²⁵²Cf Oxide Cermet Wire

TABLE II

Pd-²⁵²Cf Oxide Cermet Development Wires

Source	Process	μg ²⁵² Cf	μg ²⁵² Cf/inch	Dimensions, inch			Yield, %
				Length	Core	Clad	
<u>Bare</u>							
1005C	Evap Admix	805	179	4.5	0.030		69
1009C	Evap Admix	587	156	3.75	0.030		61
1014C	Chem Plate	607	135	4.5	0.030		89
1018C	Chem Plate	735	397	1.85	0.044		73 ^a
1020C	Chem Plate	38	11	3.4	0.040		93
1021C	Chem Plate	26	6	4.3	0.030		95
1022 3C	Chem Plate	711	426	1.67	0.037		71 ^b
		3509					
<u>Roll Clad</u>							
1005C		245	36	6.8	0.013	0.048	
1014C		43	57	0.75	0.019	0.083	
		51	13	4.0	0.010	0.042	
1021C		10	1	8.3	0.015	0.062	
		4	1	3.0	0.015	0.062	
		353 ^c					

^a No carrier

^b Powder lost during fabrication of billet

^c Included in total for bare wire

PRODUCTION OF CERMET BY
CHEMICAL PLATING

²⁵²Cf Feed

²⁵²Cf used in preparing palladium-²⁵²Cf oxide cermet was produced by irradiating plutonium, americium, and curium in high flux reactors at Oak Ridge and Savannah River. ²⁵²Cf presently is being separated at Oak Ridge National Laboratory (10). Bulk quantities are shipped to SRL in platinum alloy containers containing ²⁵²Cf oxysulfate as a residue from calcined cation resin. Assayed quantities of ²⁵²Cf are supplied for wire fabrication

as nitrate solution or as oxide (3,5). Feed specifications require that total actinide and lanthanide impurities not exceed the californium content and non-radioactive cations not exceed four times the californium content (3). Major nonradioactive impurities are calcium, sodium, potassium, barium, magnesium, aluminum, copper, iron, zinc, and lead as determined by spark source mass spectrographic analysis of ²⁵²Cf feed. Isotopic purity of the californium is typically 79% ²⁵²Cf, 15% ²⁵⁰Cf, 4% ²⁵¹Cf, and 2% ²⁴⁹Cf.

Process Solutions

The ²⁵²Cf feed is leached from the product capsule with ~25 ml of 0.1N HNO₃ into a Pyrex* process vessel (Fig. 3).

Fig. 3 Chemical Plating Process Vessel during Coating of Oxalate Particles. Flask contains Pd(NH₃)₄(NO₃)₂ solution being added dropwise. Vessel is rotated to right to filter coated particles.

* Trademark of Corning Glass Works, Corning, N. Y.

For <2 mg of ^{252}Cf , terbium carrier as 10 mg of terbium nitrate in 20 ml of distilled water is also added to the process vessel if it was not included with the ^{252}Cf during preparation of the product capsule. Because of the low cross section for neutron capture by terbium, gamma rays from activated terbium will not noticeably interfere with use of cermet sources in activation applications. For each milligram of ^{252}Cf , forty milligrams of oxalic acid in ~50 ml of distilled water is added to the vessel, and the contents are stirred for ~30 minutes to ensure complete ^{252}Cf precipitation. This quantity of oxalic acid is about a four-fold excess for precipitation of all the terbium carrier and 1 mg of ^{252}Cf and its associated oxalate-forming impurities, but is not sufficient to cause precipitation of palladium oxalate to interfere with the plating step. Oxalate agglomerates tend to form, but disperse into a cloudy suspension when the hydrazine hydrate reductant is added. Such dispersion into small particles is desirable because it promotes better ^{252}Cf uniformity in the cermet.

Chemical Plating with Palladium

Cermet is generally produced in batches requiring about 1 g of palladium. About 10 ml of 85% hydrazine hydrate is added to the ^{252}Cf -containing suspensions; this is sufficient to reduce 1 g of palladium ion to palladium metal. Palladium tetrammine dinitrate plating solution with 1 g of palladium per 100 ml of distilled water is added dropwise to the process vessel while the oxalate particles are kept suspended by stirring. The hydrazine hydrate reduces the palladium ion to palladium metal, which coats the oxalate particles. Although a thin metallic coating forms on the walls of the process vessel and the stirring bar, this coating peels off during stirring and is processed along with the palladium-coated oxalate particles. When coating is complete, the particles settle out of solution rapidly, leaving a clear supernate. The process vessel then is rotated to orient

the glass filter frit down, and the liquid is removed by vacuum filtrating, leaving the coated particles on the frit. The powder is rinsed free of excess oxalic acid and hydrazine hydrate with 200 to 500 ml of distilled water.

Drying and Calcining to Cermet

A furnace is positioned about the filter, and the coated powder on the frit is dried by heating at 200°C in argon until all traces of moisture in the process vessel have disappeared. The dried powder is pulverized on the frit and heated at 450°C in 4% H_2 -96% He for about thirty minutes to calcine the oxalate to oxide. Hydrogen in the palladium must be removed by converting from 4% H_2 -96% He to argon on cooling below 200°C, because the hydrogen will react exothermally with atmospheric oxygen and cause some of the palladium to oxidize. The cermet filter cake is again pulverized before handling.

The calcined cermet powder consists of submicron oxide particles containing californium embedded in a sponge-like palladium matrix (Fig. 4). Multipoint B.E.T. surface area analysis of calcined cermet powder using low temperature adsorption of nitrogen indicated a surface area of 3 to 8 m^2/g corresponding to an equivalent spherical particle diameter of 600 to 2000 Å. Agglomerates of freshly calcined cermet powder are free-flowing, but behave like fine lamp black when smeared. Prolonged exposure to the atmosphere causes the powder to stick from adsorbed moisture; therefore, such powder must be redried before handling.

Fig. 4 Agglomerate of Calcined $\text{Pd-Sm}_2\text{O}_3$

Powder Metallurgy Fabrication of Cermet Pellets

Cermet powder is fabricated into desired source shapes by powder metallurgy techniques. Pellets with >90% density are made by pressing the calcined cermet powder into the form, for example, of a right cylinder, ~ 0.25 inch in diameter and ~ 0.25 inch long, and sintering at 1300°C for two hours. A pressing pressure of 14,000 psi produces acceptable pellets; higher pressures (e.g., 40,000 psi) produce density variations which result in laminations in the sintered pellets. A slow two-hour heatup to the 1300°C sintering temperature allows outgassing to occur before the porosity in the pellet closes. Oxidation of the porous palladium is avoided during heatup by using 4% H_2 -96% He atmosphere up to 1000°C and then converting to argon for sintering.

Sintered pellets containing less than 2.5 vol % total oxide can be fabricated into wire by standard methods such as rolling, swaging, and drawing. However, lower density sintered cermet pellets containing up to 40 vol % oxide are durable enough to withstand normal handling as pellets. Thus, palladium- ^{252}Cf oxide cermet pellets containing up to ~ 10 mg of $^{252}\text{Cf}/\text{g}$ of palladium can be pressed and sintered for direct encapsulation into sources for neutron source applications.

Fabrication of Wire by Rolling

Sintered cermet pellets containing up to 2.5 vol % total oxide are rolled into wires routinely with square cross sections down to 0.030 inch on a side using a modified and automated 12-groove Model 27B Newton rolling mill. A high-torque reversible slow speed drive facilitates remote control for in-cell operation. Each groove produces a 30 to 40% reduction in area from the previous one. After rolling through each groove, the cermet wire is annealed in flowing argon at 850°C for 10 minutes.

Splintering generally occurs during rolling to small cross sections of cermet wire with ≥ 5 vol % oxide. Fuschillo and Gimpl (9) experienced similar difficulty in fabricating dispersion-hardened gold and platinum with ≥ 2 vol % oxide. Besides the terbium carrier added, impurities in the ^{252}Cf feed equivalent to five times by weight the californium content can produce an oxide volume up to ten times the volume of californium oxide. Thus, the linear ^{252}Cf concentration in Pd- ^{252}Cf oxide cermet wire will vary as a function of wire size and ^{252}Cf loading as shown in Fig. 5.

The microstructure of Pd- Sm_2O_3 cermet wire, which is probably typical of that in Pd- ^{252}Cf oxide wire with maximum ^{252}Cf concentration, is shown in Fig. 6.

Fig. 5 Linear ^{252}Cf Concentration in Cermet Wire versus ^{252}Cf Loading

Fig. 6 Palladium-2.5 Vol % Sm_2O_3 Cermet Wire

Rolling has been used to apply a palladium cladding to bare cermet to further reduce the linear ^{252}Cf concentration or provide containment of surface contamination (Table II). Bare cermet wire is inserted into Pd tubing, rolled to form a square sheath, and rolled to a precalculated length to reduce the linear ^{252}Cf loading to the desired level (Fig. 7).

Fig. 7 Sheathed Cermet Wire

Uniformity of ^{252}Cf distribution is maintained except for an end defect about equal to the core diameter. Fabrication of three inches of clad cermet wire containing $\sim 4 \mu\text{g}$ of ^{252}Cf ($\sim 6 \times 10^9 \alpha \text{ d/m}$) with a contamination-free surface was demonstrated without contaminating the equipment. This clad wire was subdivided in three places with no detectable smearable contamination ($< 20 \alpha \text{ d/m}$) using a pinch-cutting tool which separated the core while

maintaining the containment in the sheath (Fig. 8).

Fig. 8 Pinch Cutting of Roll Clad Cermet Wire

Sheathed cermet wire fabricated by a combination swaging-drawing method has been fabricated with Pd- Sm_2O_3 cermet (11). A sintered cermet pellet is coined and welded into a sheath of palladium alloy with a hardness somewhat greater than the cermet core. The assembly is swaged to ~ 0.100 inch in diameter and drawn through a series of dies, each reducing the cross section area by $\sim 10\%$. Pd- Sm_2O_3 cermet containing up to 2 vol % oxide has been drawn down to 0.010-inch-diameter wire with a 0.004-inch cermet core. Wires of this size are being used as neutron sources for medical applications (12).

Uniformity of ^{252}Cf Distribution

The ^{252}Cf content of Pd- ^{252}Cf oxide cermet wires is assayed within $\pm 3\%$ by neutron counting. Uniformity of the linear ^{252}Cf distribution in wires is determined by autoradiography with Kodak Spectrum Analysis No. 1 plates that are sensitive to soft gamma rays. A 0.025-inch-thick stainless steel sheet inserted between the plate and wire during exposure improves resolution over a direct exposure. Exposure time decreases with increasing ^{252}Cf concentration from ~ 2 minutes for $5 \mu\text{g/inch}$ to ~ 2 seconds for $500 \mu\text{g/inch}$. Automated densitometer measurements are made on exposed plates

at 1 mm intervals and converted to percent absorption from which an average and deviations from the average for each interval are computed. The decrease in exposure at the ends of a wire precludes analysis of uniformity within 1/8 inch of the ends. Comparisons of autoradiographic results with ^{252}Cf distribution determined by neutron counting of measured and cut wire sections confirm that autoradiography can reliably assess uniformity with a precision of $\pm 2\%$. Autoradiography of Pd- ^{252}Cf oxide cermet wires produced in the demonstration program revealed that ^{252}Cf in each 1 mm segment of wire is generally within $\pm 10\%$ of the average concentration (Fig. 1). Deviations from uniformity are thought to result mainly from variations in wire cross section rather than inhomogeneous distribution of ^{252}Cf in the cermet.

Recovery

A nitric acid-anion exchange process was demonstrated for recovery of ^{252}Cf from Pd- ^{252}Cf oxide cermet. Hot concentrated nitric acid with a trace of hydrochloric acid is used to dissolve the cermet. The solution is adjusted to ~ 20 ml volume with 0.1N acidity and passed through a room temperature column containing 100 ml of *Dowex 1-X8** (200 to 900 mesh) resin by gravity flow at 3 ml/minute to extract the californium. The californium is recovered by elution with 6N nitric acid. The important long-lived ^{248}Cm produced by alpha decay of ^{252}Cf can be separated from the ^{252}Cf following denitration of the solution by pressurized elution development chromatography with alphas-hydroxyisobutyrate (13).

^{252}Cf Losses

Table II summarizes the process yields during fabrication of developmental Pd- ^{252}Cf oxide cermet wire. The chemical plating process is expected to result in cermet wires containing $\geq 90\%$ of the invested ^{252}Cf . Low yields attributed to lack of

terbium carrier and mishandling of powder are not expected to occur during full-scale production. Typically, $\leq 10\%$ of the ^{252}Cf will be in splintered wire ends; $\leq 10\%$ will remain in the leached supply capsule, chemical plating apparatus, and filtrate; and $\leq 5\%$ will be lost during pellet and wire fabrication. Only losses in the product capsule and filtrate affect the californium/palladium ratio so that the ratio can be fabricated within 95% of the desired composition. The other losses affect the californium/palladium ratio and wire length equally. Only the ^{252}Cf remaining in the supply capsule is irrecoverable; the rest can be processed for recovery as described previously.

PROPERTIES OF Pd- ^{252}Cf OXIDE CERMET

The chemical and physical properties of Pd- ^{252}Cf oxide cermet are those of palladium and a mixed oxide of californium, certain feed impurities, and terbium carrier. The major feed impurities that are incorporated into cermet are the actinides and lanthanides, barium, calcium, copper, iron, zinc, and lead.

Superficial oxidation of cermet wire will occur at 400 to 800°C, but not at higher temperatures, because palladium oxide is unstable over 800°C. Significant corrosion occurs only in strongly oxidizing chemicals, such as nitric acid, hot sulfuric acid, ferric chloride and hypochlorite solutions, chlorine, bromine, iodine, and hydrogen sulfide (at $> 600^\circ\text{C}$). No reactions are expected between Pd- ^{252}Cf oxide cermet and stainless steels or zirconium up to 1000°C, nor have any reactions been observed between the palladium matrix and the oxide phase up to 1300°C.

The melting point of Pd- ^{252}Cf oxide cermet is expected to be close to that of pure palladium (mp = 1552°C). Volatility of californium oxide is expected to be insignificant up to 1000°C, where the oxide vapor pressure is estimated to be $\sim 10^{-12}$ torr. No transport of ^{252}Cf from cermet has been observed during two hours of sintering

* Tradename of Dow Chemical Co., Midland, Mich.

at 1300°C in argon or while the cermet was molten for 15 minutes at 1600°C in 4% H₂-96% He.

Neutron and gamma spectra from Pd-²⁵²Cf oxide cermet wire sources are similar to those from oxide sources in platinum-rhodium capsules (3). The gamma spectrum has considerably fewer large peaks than for oxide sources encapsulated in stainless steel capsules.

The mechanical properties of Pd-²⁵²Cf oxide cermet wire are expected to be the same as those measured for Pd-Sm₂O₃ cermet wire with equivalent total oxide contents. Elongations of 3 to 14% are to be expected (Fig. 9). The ultimate tensile strength should vary between 20,000 and 40,000 psi depending on the total oxide content of cermet (Fig. 10). Yield strengths between 9,000 and 17,000 psi are typical and are not affected significantly by oxide concentration (Fig. 11). Hardness will increase with oxide content from about 50 DPN for pure palladium to about 80 to 100 DPN for annealed cermet wire with up to 7.5 vol % oxide (Fig. 12), but cold working can further increase cermet hardness up to 200 DPN.

Fig. 9 Ductility of Pd-Sm₂O₃ Cermet

Fig. 10 Ultimate Strength of Pd-Sm₂O₃ Cermet

Fig. 11 Yield Strength of Pd-Sm₂O₃ Cermet

Fig. 12 Hardness of Pd-Sm₂O₃ Cermet

EFFECT OF DECAY HELIUM ON WIRE INTEGRITY

The embrittling effect of decay helium on Pd-²⁵²Cf oxide cermet is not expected to be significant for the ²⁵²Cf concentrations and lifetimes required. A reduction in ductility, especially during or after high temperature anneals, has been observed in other materials when the volume of entrapped gas exceeds a few tenths of the sample volume (14). Similar volumes of helium

will be generated in about a year in Pd- ^{252}Cf oxide cermet containing 2000 μg of ^{252}Cf per gram of palladium (Fig. 5). Effects of decay helium on cermet fabricability and mechanical properties were assessed using ^{242}Cm (163-day half-life) as a stand-in for ^{252}Cf . A 0.060-inch Pd- $^{242}\text{Cm}_2\text{O}_3$ cermet wire, which had been stored for two months to accumulate a helium content equivalent to a volume 0.6 times the cermet wire volume, was successfully reduced to 0.040-inch cross section by rolling and annealing. However, after further storage for five months to allow the helium content to increase to 1.8 times the wire volume, the wire was noticeably embrittled and snapped in half when slightly bent; Pd-Sm $_2\text{O}_3$ cermet wire with equivalent oxide content could be easily bent into a coil with $\sim 1/16$ -inch radius. Annealing at 850°C restored ductility to the embrittled Pd- $^{242}\text{Cm}_2\text{O}_3$ cermet wire such that it could be bent without breaking. Thus, embrittlement of Pd- ^{252}Cf oxide cermet is not expected to affect initial fabrication or reshaping of aged sources after annealing.

CONCLUSIONS

Palladium- ^{252}Cf oxide cermet containing up to 2 mg of ^{252}Cf per gram of palladium can be fabricated into convenient shapes for use as neutron sources. Since less expensive containment facilities may be required for encapsulation of cermet neutron source forms than for forms requiring quantitative wet chemical operations, the cost of finished sources may be significantly lower than present oxide sources.

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MacMurdo, M. A. Wakat, B. Tiffany, and R. H. Gaddy developed autoradiography and automated densitometer assessment of ^{252}Cf uniformity in wires. Dr. W. R. McDonnell assessed the effect of decay helium on cermet integrity. R. M. Henry measured the mechanical properties of the cermet wires. B. L. Dahlen developed the wire rolling techniques and selected palladium as a matrix material. Dr. S. F. Peterson and T. R. Herold measured the gamma and neutron spectra of cermet. S. D. Fulbright demonstrated the freeze-drying admixing technique.

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