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OPTIMIZATION OF MELTING PARAMETERS FOR FABRICATION OF (U-Pu)C

AUTHOR

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STATEMENT OF PROBLEM			
Determine a system of melting and casting that will reduce the loss by volatilization, of plutonium,during the fabrication of (U-Pu)C.			
ABSTRACT			
A theoretical study on the loss, by volatilization, of Pu from (U-20w/oPu)C was completed and indicated that the loss rate would be reduced by a factor of 3000 by melting in one atmosphere of argon as opposed to melting in a vacuum. Studies of crucible geometry (without a heat transfer analysis) indicated that a hemispheric crucible would be an optimum design to minimize the amount of skull within the crucible. Heat transfer studies, partially completed, may disqualify the hemispheric shape due to the extremely large heat loss from the open crucible top which would necessitate extremely large power inputs, possibly long melting times and consequently larger Pu losses.			

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I INTRODUCTION

Thousands of kilograms of uranium carbide have been arc melted and cast into slugs for use in nuclear reactor fuel elements. It is now desired to utilize the arc melting and casting technique to produce mixed, (U-Pu)C. However the plutonium fraction addition presents a much more formidable problem due to its toxicity, high vapor pressure relative to uranium, high specific radioactivity, and high cost, \$43/gm compared to U^{235} at \$12/gm. In addition, the safe batch size (safe batch size is defined as 45% of a critical mass under optimum conditions to produce criticality) for plutonium is only 71% of the safe batch for U^{235} . However, arc melting and casting is an attractive technique to fabricate fuel slugs for a fast breeder reactor due to the directness in producing fabricated shapes from raw materials. It is an ideal technique to produce high density material, approximately 98%, of the theoretical density of the composition involved.

The first problem to be solved is the problem of the loss of plutonium, by volatilization, during the melting process. Various researchers have reported losses from 1 to 50% of the contained plutonium during the arc melting operation. The effect of the loss has a pronounced economic effect upon fabrication. As an example, a 1% non-recoverable loss of Pu per kilogram of (U-20w/oPu)C would result in an additional cost of \$82.70/kg of fuel. The magnitude of the problem is much more evident, when compared with losses experienced in the fabrication of hyperstoichiometric carbide for a physics fuel experiment for the HWO CR program as reported in TI-010-28-013. A total non-recoverable loss of 4.08% of the incoming uranium (1,595 kg) was experienced thru the entire fabrication process from carbothermic reduction of UO_2 , arc melting and casting into slugs and machining of the cast slugs to finished size. Of the 4.08% loss, 0.6% was attributable to the carbothermic reduction process and the balance to the arc melting and machining operations. The loss from these two operations cannot be separated since powders

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were deposited in a common receptacle from a vacuum cleaning system. However, a total of 6.61% of the uranium into the casting operation ended up as dust, only part of which was recovered. This analysis emphasizes the arc melting loss problem when it is realized that the vapor pressure of Pu in the composition (U-20w/oPu)C is approximately ten times greater than the vapor pressure of uranium at the same temperature in UC. It is therefore evident, that a technique must be developed to reduce plutonium volatilization.

Volatilization of plutonium in (U-Pu)C is dependent on the concentration, temperature, exposed surface area, time and impedance to flow of gaseous species above the molten surface. Attention, during the past fiscal year, has been centered upon increasing the impedance to the flow of escaping material above the molten surface and to optimizing the geometry of the container holding the molten pool. Three studies have been performed: (1) A theoretical study of the effect of inert gas atmosphere melting upon the suppression of plutonium volatilization (2) A crucible geometry study to indicate directions for crucible design development and (3) A computer case has been written, utilizing an existing code to obtain a two dimensional heat transfer analysis to obtain a better understanding of the heat flow within the crucible during the melting process so that the amount of molten carbide within the copper skull melting crucible may be maximized. The design of the crucible will also, however, be a compromise between maximizing the pool size and minimizing the plutonium loss by volatilization. The final size of the crucible will be dependent on the allowable plutonium content per melt, obtained by using the melting system parameters in a criticality calculation.

The second problem to be solved in the program is to increase the capacity of the arc melting operation. Due to the relatively high fissile content, the allowable safe batch size in the melting crucible and furnace will be severely limited. A cursory examination has indicated a maximum batch size of approx. 4000 gm of (U-20w/oPu)C. Therefore, a furnace will be designed,

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for semi-continuous operation, in which the charging of feed material and withdrawal of cast slugs will be possible without opening the furnace to the glove box atmosphere after each heat. This technique will allow for greater furnace utilization since the melting and casting period is only about 17% of the total operational time for a batch arc melting furnace. The remainder of the time is needed to cool the slugs sufficiently so that they may be removed from the furnace and to recharge the furnace. By allowing cooling outside the main arc melting chamber, and incorporating an automatic feed system, furnace utilization should increase approximately three times.

II CALCULATED VAPOR PRESSURES AND LOSS RATES OF Pu FROM UC-PuC FUEL

A. Results

The results indicate that the addition of an inert gas drastically reduces the loss of Pu from the melt. At one atmosphere pressure and 3000°K, the maximum loss rate is expected to be 20 micrograms/cm²-sec with argon and 11 micrograms/cm²-sec with xenon. Since the loss rate is inversely proportional to total pressure, the use of higher pressures will further reduce the loss. Without an inert gas, the loss rate would be 60,000 micrograms/cm²-sec.

B. Method of Calculation and Detailed Results

The results and method of calculating the vapor pressure and loss rates of plutonium during melting of UC-20w/oPuC fuel follows. The results given should be considered as upper limits since each of the assumptions involved in the calculations leads to high values for the vapor pressure and diffusion coefficients. These assumptions are: (1) ideal behavior of all species in the condensed and gaseous phases; (2) a rigid sphere model for the derivation of the expression for diffusion coefficients; (3) zero concentration of Pu vapor at 1 cm from the surface of the melt; (4) the collision diameter of gaseous Pu atoms is equal to the molar volume

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of the solid divided by Avogadro's number; and (5) the vapor pressure of Pu over the melt can be obtained by extrapolation from 1900° to 3000°K of the expression obtained from observed pressures over the solid.

The desired composition of the carbide fuel is 20 w/o PuC in UC. These concentrations yield mole fractions for PuC and UC of 0.1993 and 0.8007, respectively. Olson and Mulford⁽¹⁾ recently studied the thermodynamics of plutonium carbides by measuring the vapor pressures to approximately 1900°K of the systems PuC + Pu₂C₃ and Pu₂C₃ + C. Their results may be expressed as:

Since stoichiometric PuC is almost impossible to achieve, it is assumed that the plutonium carbide in the fuel will be present as a mixture of PuC_{0.87} and Pu₂C₃ so that we have a mole fraction of PuC_{0.87} = 0.15817 and a mole fraction of PuC_{1.5} = 0.04113 rather than 0.1993 mole fraction of PuC. Hence, for our fuel, the standard free energy of the reaction

may be taken to be

$$\Delta F_3^\circ = 0.15817 \Delta F_1^\circ + 0.04113 \Delta F_2^\circ = 18179.9 - 4.281T \text{ cal} \quad (3a)$$

If we assume that the resulting carbon combines with some of the UC to form U₂C₃ we obtain

Holley and Storms⁽²⁾ give the free energy of formation at 2100° and 2300°K for uranium monocarbide and uranium sesquicarbide. Using these data and assuming that the free energy is a linear function of temperature yields

The reaction given by (4) can then be rewritten as the sum of reactions (7) and (8)

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$$\Delta F_7^\circ = 0.3986 (22630 + 1.2T) \text{ cal} \quad (7)$$

$$\Delta F_8^\circ = -0.1993 (48500 + 2.4T) \text{ cal} \quad (8)$$

to yield a free energy for reaction (4) of

$$\Delta F_4^\circ = -645.7 \text{ cal} \quad (4a)$$

Thus, neglecting vaporization of uranium, the overall reaction for our fuel is the sum of reactions (3) and (4),

The free energy of this reaction is given by the sum of the free energies for reactions (3) and (4)

$$\Delta F_{10}^\circ = 17534 - 4.28T \text{ cal} \quad (10a)$$

The equilibrium constant of the reaction, K, is defined as

$$K = (a_{\text{Pu}})^b (a_{\text{U}_2\text{C}_3})^b + (a_{\text{UC}})^c (a_{\text{PuC}})^b$$

where b and c are the coefficients of the species in equation (10), and the a's are the activities of those species. If we assume ideal behavior, the activities of the carbides will be equal to their mole fractions and the activity of the plutonium vapor will be equal to its pressure in atmospheres. Hence, since $\ln K = -\Delta F^\circ/RT$,

$$\ln (p)^b = -\Delta F^\circ/RT + n(a_{\text{UC}})^c \quad (11)$$

and, converting to natural logarithms, noting that $a_{\text{UC}} = 0.500$,

$$\log p = -19227/T + 4.092 \quad (12)$$

If we assume that the results given in references (1) and (2) may be extrapolated to 3000°K, the vapor pressure of plutonium over the fuel will be that given in Table 1. (This assumption is conservative since expressions used were obtained for solid carbides and the vapor pressure over a liquid is usually less than that over the solid at the same temperature.)

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TABLE 1

Equilibrium Vapor Pressure of Pu Over UC-20w/oPuC

Temperature (°K)	Vapor Pressure	
	(atm. x 10 ³)	(mm)
2000	0.00301	0.00229
2250	0.0352	0.0268
2500	0.252	0.1914
2750	1.260	0.954
3000	4.815	3.659

From the kinetic theory of gases the maximum rate of evaporation of Pu from the solution is given by

$$\mu = p(M \div 2\pi RT)^{1/2} = (6.766 \times 10^{-4})p \div T^{1/2} \text{ gm/cm}^2\text{sec} \quad (13)$$

where M is the molecular weight (239.127), R is the gas constant (83.143×10^6 erg/deg-mole), p is the vapor pressure in dynes/cm², and T is the temperature in °K. Substitution of the vapor pressures from Table 1 yields the results given in Table 2. It should be noted that actual loss rates should be less since the activity coefficient of PuC in the melt would be expected to be less than unity if its behavior is similar to that of UC. In addition, the equilibrium vapor pressure would be expected to be less than those given in Table 1 since these pressures were obtained by extrapolation of observed pressures over the solid carbides.

TABLE 2

Maximum Rate of Evaporation of Pu

Temperature (°K)	Evaporation Rate (mg/cm ² sec)
2000	0.0461
2250	0.509
2500	3.452
2750	16.42
3000	60.26

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Although the addition of an inert gas would be expected to have a negligible effect (less than 0.1%) on the equilibrium vapor pressure it should reduce the evaporation rate by reducing the rate of diffusion of the vapor from the surface of the solution. The kinetic theory of gases, assuming a rigid sphere model, yields the following expression for the diffusion coefficient in a binary gas mixture⁽³⁾:

$$D = (2.6280 \times 10^{-3}) (T^{3/2} / (M_1 + M_2))^{1/2} \div P \sigma_{1,2}^2 (2M_1 M_2)^{1/2} \text{ cm}^2/\text{sec} \quad (14)$$

where T = temperature in °K

M_1 = molecular weight of species 1

M_2 = molecular weight of species 2

P = total pressure in atmospheres, and

$\sigma_{1,2} = (\sigma_1 + \sigma_2) \div 2$ = the mean collision diameter in Å.

The collision diameter of the gaseous atoms of plutonium can be estimated from the density of the solid, by assuming that the atoms of plutonium in the solid occupy the entire volume. Thus, using the subscript 1 to represent Pu, and taking the density of solid plutonium as 19.85⁽⁴⁾,

$$\sigma_1 = 10^{-8} (6M \div \pi \rho N)^{1/3} = 3.37 \text{ Å}$$

where N is Avogadro's number and ρ is the density, and $M = M_1 = 239.127$ gm. Equation (14) shows that, for a given binary mixture, the diffusion coefficient will be given by

$$D = AT^{3/2} \div P \text{ cm}^2/\text{sec} \quad (15)$$

where A is a constant for the mixture and is determined from the molecular weights and collision diameters of the two species. The molecular weights and collision diameters⁽⁵⁾ of argon, krypton, and xenon given in Table 3 yield the constants, A, for use with equation 15. The corresponding diffusion coefficients for a total pressure of one atmosphere are given in Table 4. Since the rigid sphere model assumes that there are no inter-molecular forces, these diffusion coefficients are undoubtedly higher than those which will occur in practice, and may therefore be considered as upper limits.

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TABLE 3

Constants for the Evaluation of Diffusion Coefficients

Gas	Atomic Weight (gm)	Collision Diameter $\bar{A}$	A (see eqn. 15)
Argon	39.948	3.465	2.715×10^{-5}
Krypton	83.30	3.61	1.941×10^{-5}
Xenon	131.30	4.055	1.464×10^{-5}

TABLE 4

Diffusion Coefficients for Pu-Inert Gas Mixtures

Temperature °K	Diffusion Coefficient (cm ² /sec)*		
	Argon	Krypton	Xenon
2000	2.428	1.736	1.310
2250	2.897	2.072	1.563
2500	3.393	2.426	1.831
2750	3.915	2.799	2.122
3000	4.461	3.189	2.406

*Divide the tabulated values by the pressure in atm.
to obtain the diffusion coefficient at any total
pressure.

The loss of plutonium from the solution due to isothermal
diffusion with a concentration gradient expressed as the change
in the partial pressure of plutonium per centimeter of distance
from the surface of the solution is given by ⁽⁶⁾

$$\mu = DM(p_1 - p_2) \div RT \quad \text{gm/cm}^2 \text{sec} \quad (16)$$

where D = diffusion coefficient (cm²/sec)

R = gas constant (ergs/deg mole)

p₁ = partial pressure at the surface of the solution (dynes/cm²)

p₂ = partial pressure 1 cm from the surface of the solution
(dynes/cm²)

Hence, for the three mixtures being considered

$$\mu_A = 7.911 \times 10^{-5} T^{1/2} (p_1 - p_2) \div P \quad \text{gm/cm}^2 \text{sec} \quad (17a)$$

$$\mu_{Kr} = 5.657 \times 10^{-5} T^{1/2} (p_1 - p_2) \div P \quad \text{gm/cm}^2 \text{sec} \quad (17b)$$

$$\mu_{Xe} = 4.268 \times 10^{-5} T^{1/2} (p_1 - p_2) \div P \quad \text{gm/cm}^2 \text{sec} \quad (17c)$$

For the extreme case of zero concentration at 1 cm from the surface of the solution, $p_2 = 0$ so that the maximum loss at one atmosphere total pressure will be that given in Table 5. It should be noted that the addition of one atmosphere of argon reduces the loss rate from $60 \text{ mg/cm}^2\text{sec}$ to $0.02 \text{ mg/cm}^2\text{sec}$ --- a reduction by a factor of nearly 3000; xenon is approximately twice as effective as argon in reducing the loss rate.

TABLE 5

Evaporation Rate of Pu in Presence of Inert Gases*

Temperature °K	Equil. Pressure of Pu (atm)	Evaporation Rate ($\mu\text{g/cm}^2\text{sec}$)		
		Argon	Krypton	Xenon
2000	3.01×10^{-6}	0.011	0.008	0.006
2250	3.52×10^{-5}	0.132	0.094	0.071
2500	2.52×10^{-4}	0.996	0.712	0.537
2750	1.26×10^{-3}	5.23	3.74	2.82
3000	4.82×10^{-3}	20.9	14.9	11.3

*The tabulated rates are for 1 atm. total pressure. For other pressures divide these values by the total pressure in atmospheres.

III CRUCIBLE GEOMETRY STUDY

An examination was made of the analytical parameters involved in optimizing a crucible design for arc melting mixed carbide fuel. The problem was defined as maximizing the net output of molten mixed carbide, starting with a maximum allowable mass of Pu in the crucible. The fixed parameters include the use of a liquid cooled metal crucible.

The cooled walls of the crucible cause a solidified skull to form, made up of (U,Pu)C. The plutonium contained in the skull must be counted in the maximum allowable mass of Pu for criticality control. Since the skull remains in the crucible during all subsequent melting batches, the problem becomes one of minimizing the mass of skull remaining to maximize the amount of charge added for each batch and, consequently, the net molten

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mixed carbide output.

The mass of the remaining skull is found from the product of the surface area (crucible walls) times the integrated thickness times the density of the skull. Assuming a constant density, the obvious solution would be to minimize the skull thickness and surface area. Granting that the skull-molten interface occurs at a constant temperature and that the crucible wall is at a constant temperature, the skull integrated thickness can be minimized by shaping the crucible walls to coincide with the skull-molten isothermal line. The skull thickness can also be decreased by increasing the crucible surface temperature and/or increasing the temperature gradient through the skull (increase q/A - heat flux).

Turning next to the area considerations, there are two incentives for decreasing the crucible surface area while maintaining the allowable volume of $(U,Pu)C$. First, the volume of skull will decrease with an area decrease. Secondly, a decrease in surface area will increase the heat flux (q/A) through the skull for a given q , aiding in reducing the skull thickness by raising the temperature profile through the skull. The sphere has the most optimum volume to area ratio for closed shapes, but the vessel must have an open top. A hemisphere has the least wall surface area for a fixed volume in an open top vessel, and a cylinder with the diameter equal to twice the height ($D = 2H$) is the next best shape.

A hemispherical crucible would satisfy three requirements for decreasing the skull volume. The first is to minimize the surface area of the skull as related to the total $(U,Pu)C$ volume. Secondly, the isothermal lines associated with a point heat source in a semi-infinite solid (infinite depth and width with a top surface) radiate from the source in hemispherical fronts. This causes the crucible walls and the isothermal lines to coincide, which decreases integrated skull thickness. The third reduction occurs by minimizing the skull thickness when the heat flux (q/A) increases with the area decrease.

The disadvantage of a hemisphere is the large open surface area, which would allow greater convective, radiant and volatile

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heat losses. These losses must be balanced against the gains achieved in reducing the crucible wall surface area. The following table summarizes the crucible wall area, diameter and open top surface area for unit volumes of three cylindrical shapes and a hemisphere.

Shape	Diameter	(1) Wall Area	(2) Top Area	(2)÷(1)	Volume÷(1)
Hemisphere	1.56	3.82	1.91	0.50	0.26
Cylinder					
D = 2H	1.36	4.39	1.45	0.33	0.23
D = H	1.08	4.63	0.92	0.20	0.22
D = H/2	0.86	5.21	0.58	0.11	0.19

The above discussion assumes the (U,Pu)C charge acts like a semi-infinite solid in the heat transfer mechanism. Since the dimensions of the crucible are relatively small, the heat transfer will depart somewhat from this ideal case. Nevertheless, it is felt that the hemisphere will serve best in reducing the skull volume. On the other hand, the losses from the large open top of the hemisphere may be too large, and necessitate a compromise between this shape and a cylinder. The table indicates the relative area values for the different shapes, and an idea of the trade-offs can be obtained.

IV CRUCIBLE HEAT TRANSFER STUDY

No definitive data has been obtained from the crucible heat transfer study. A computer case utilizing an existing code for heat transfer was prepared and partially tested. Difficulty was experienced with the temperature iteration equations within the program which diverged for certain sets of input parameters. Additional work will be performed on the heat transfer study next fiscal year when additional funds will be available.

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