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A THERMODYNAMIC DATA PROGRAM INVOLVING PLUTONIA AND URANIA AT HIGH TEMPERATURES

QUARTERLY REPORT NO.14

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November 1, 1970 - January 31, 1971

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ABSTRACT

Work progress is reported in the continuing scope of work related to vaporization of urania in urania-plutonia solid solutions, oxygen activities at high temperatures, oxygen redistribution in a temperature gradient, Pu/U segregation and sodium-fuel interactions.

1. INTRODUCTION

This quarterly report is the fourteenth in a series which describes experiments directed toward understanding plutonia segregation in urania-plutonia solid solutions and in fast reactor environments through a knowledge of its thermodynamic behavior. In the thirteenth quarterly (GEAP-12153) the progress of several experiments which have a bearing on plutonia segregation was described. In addition the thermodynamic aspects of the sodium-fuel reaction product were studied. Continued progress in these areas is described in this report.

2. THERMAL GRADIENT EXPERIMENTS WITH EXCESS SODIUM

In the twelfth quarterly report, an experimental system was described in which a stack of fuel pellets of a given oxygen-to-metal ratio was exposed to sodium in a sealed molybdenum capsule. The molybdenum capsule was heated in a temperature gradient so that reaction between sodium and fuel would be free to occur at the point of maximum stability. Preliminary results indicated the reaction compound formed near, but not at, the coldest end of the capsule when heated in the temperature range of 550 to 1150°C. A second series of capsules showed the same tendency when heated between 900 and 1450°C. These findings were similar to the situation found in failed irradiated fast reactor fuel capsules where the sodium reacts only with fuel near the outer periphery where it is cooler.

By varying certain key parameters it is possible to determine from this type experimental system, the nature and thermodynamic conditions for reaction of sodium with the fuel. Such a study has been initiated with the objective of determining the threshold conditions for reaction.

The sodium-fuel reactions may be described as follows:

where it is assumed the sodium-fuel compound is $\text{Na}_3(\text{U,Pu})\text{O}_4$. The extent of reaction in the presence of excess sodium is determined by the difference in y and x , which, respectively, are measures of the final and initial oxygen-to-metal ratio. The value for y under equilibrium conditions is a function of temperature and activity of sodium. Under reactor conditions with failed fuel the sodium phase would not be necessarily at the same temperature as the reaction product phase; furthermore, the fuel phase exists over a large temperature gradient. Therefore, conditions are most meaningful when they are obtained in the presence of a temperature gradient.

The sodium phase should condense at the coldest end of the capsule where its temperature, T_N , determines the vapor pressure of sodium in the system. The chemical potential for sodium at some position, i , in the capsule is given by (2)

$$\mu_i(\text{Na}) = R T_i \ln P_{\text{Na}} \quad (2)$$

where P_{Na} is the vapor pressure of sodium and T_i is the temperature at position, i . The reaction compound may form over a temperature interval or at one specific point. We define T_R as the maximum temperature of this interval. At $T > T_R$ the MO_{2-x} fuel phase is the only condensed phase present. In the free space of the capsule, sodium vapor is present and is free to interact with the fuel phase and allow oxygen to migrate as gaseous NaO or Na_2O to the low temperature regions where it may form the reaction product. Given sufficient time to establish steady-state conditions the reaction point at T_R will be adjacent to the fuel phase at a composition of MO_{2-y} . Therefore, this composition is the threshold value for formation of the reaction compound. The variation of the oxygen-to-metal ratio of the fuel at $T > T_R$ will be characterized by the heat of transport, Q^* . The heat of transport as described previously¹ is established by the mechanism(s) of oxygen transport in the gas and solid phase of the system.

At T_R , if the initial fuel composition, $(2-x)$ is less than the threshold value, $2-y$, then no reaction would occur. However with time as oxygen migrates to the low temperature end, and if $2-x$ reaches the threshold value at T_R then reaction would occur directly with sodium vapor without oxygen migration. The amount of reaction compound depends on the initial average oxygen-to-metal ratio of the fuel phase, if it is assumed there is an excess of sodium.

As a result of the nonisothermal situation, the two independent parameters, T_N and initial oxygen-to-metal may be varied systematically to determine the conditions for reaction. T_R , y at T_R , and Q^* are dependent parameters obtained from the experiment. The system is constrained to be at steady state and to contain an excess of sodium. The time for steady-state conditions is established by experiment. The temperature gradient experiments in excess sodium performed so far are given in Table 1. Except for capsule 5, all experiments showed visual evidence of excess sodium after the heat treatment. It is surmised this capsule developed a leak during heat treatment and the sodium was lost. Capsule 1 was run for a short time at a low temperature with stainless steel as the capsule material to demonstrate sodium would migrate to the cold end and possibly produce a reaction compound. Reaction product was evident at 750°C, but migration of oxygen from the high temperature end was insufficient for the time and temperatures used in this experiment to establish steady-state conditions.

TABLE 1. Summary of Experimental Conditions and Results from Thermal Gradient Capsules Heated in a Temperature Gradient

Capsule	Internal Temperature (°C)	Heat Treatment (h)	Na (mg)	Initial O:M	T_N (°C)	T_R (°C)	2-y at T_R	Q^* (kcal/mole)	Remarks
1	500-1150	100	35	1.965	500	750	1.965	NM [†]	Not at steady state
2	900-1450	300	32	1.965	900	975	1.970	-5.7	Reaction bands < 1000°C
3	900-1450	300	85	1.965	900	980	1.975	-5.7	Reaction bands < 1000°C
4	1105-1475	300	37	1.933	1110	1110	1.94-1.95	-2.4	Reaction at coldest pellet only
5	1240-1500	300	36	1.964	1240	—	NM	-15.7	No reaction product, no visual evidence of Na Capsule may have failed.
6	1105-1475	100	39	1.958	1060	~1100	NM	—	1 inch long pellet at cold end. Not at steady state
7	1105-1475	1000	49	1.966	—	—	—	—	Examination incomplete
8	1105-1475	300	138	1.80	—	—	—	—	Examination incomplete 100% $PuO_{1.8}$

[†] NM = not measured

In subsequent experiments molybdenum was used as the capsule material so that higher temperatures (900 to 1500°C) could be used to establish steady state. Capsules 2 and 3 were run simultaneously for 300 hours with two different amounts of sodium to determine the amount of sodium required at an initial oxygen-to-metal of 1.965. The results reported in previous reports in this series revealed that 32 mg of Na was sufficient to maintain an excess amount of sodium. Identical heats of transport were obtained and the reaction product formed at identical locations in the two capsules with a T_R of about 1000°C. The oxygen-to-metal values at T_R were essentially the same, 1.970 and 1.975. These two experiments indicated reaction behavior could be duplicated and the results used to predict reaction threshold conditions.

Additional capsules are being run at different conditions to assess the effect of time, sodium temperature, T_N and initial oxygen-to-metal on the reaction. Capsule 4 which contained pellets with an initial oxygen-to-metal of 1.933 yielded a heat of transport of -2.4 kcal/mole (Figure 1). Spots of metallic sodium were observed in some of the pellets after the 300-hour anneal. These pellets so contaminated were not analyzed for oxygen-to-metal because of the interference of sodium in the oxygen-to-metal determination. Metallic sodium was also present on the low temperature end of the coldest pellet. The cold end of this pellet had increased in diameter to the extent of approximately 50 mils, an indication of the presence of a reaction product between the fuel and sodium. The reaction product was preserved by storing the pellet in an inert atmosphere. The product was later examined by x-ray diffraction of the surface *in situ*. The x-ray diffraction data are shown in Table 2. The major phase² was $\text{Na}_3(\text{U,Pu})\text{O}_4$ with a lattice parameter $a_0 = 4.81 \pm 0.020 \text{ \AA}$. A minor UO_2 -25% PuO_2 solid solution phase with a lattice parameter $a_0 = 5.452 \pm 0.009 \text{ \AA}$ was also present. Questionable agreement was noted³ with $\text{Na}_{11}\text{U}_5\text{O}_{16}$. The reaction compound was deteriorating in air while on the diffractometer; therefore, attempts to detect the reaction product directly from failed irradiated fuels may require a hot cell with an inert atmosphere.

No trace of sodium was found upon opening Capsule 5. The heat of transport was -15.7 kcal/mole (Figure 2) which was similar to experimental values obtained in thermal gradient capsules without sodium. It is assumed from these observations that the capsule developed a leak and the sodium was lost. This capsule was run at a high T_N (1240°C) to determine if the reaction compound would form at $T > 1240^\circ\text{C}$. The sodium vapor pressure would have been about 10 atm which may have hastened the leak.

Capsules 6 and 7 were annealed for 100 and 1000 hours, respectively, to determine the effect of time on the oxygen redistribution and the reaction. In the 100-hour test the observed heat of transport was -10.2 kcal/mole (Figure 3) as opposed to -5.7 for 300 hours at a similar stoichiometry. Below about 1260°C, the pellets begin to exhibit a greater oxygen deficiency and approach their original value at the lowest temperature. This behavior indicates steady-state oxygen transport was not achieved in 100 hours. Examination of the 1000 hour anneal (Capsule 7) has not been completed.

FIGURE 1. ARRHENIUS PLOT OF 300 HOUR THERMAL GRADIENT ANNEAL OF $\text{U}_{175}\text{Pu}_{125}\text{O}_{1.933}$ WITH 36.88 mg SODIUM

TABLE 2. Interaction Phases on Surface of
25 wt % PuO₂-UO₂ Pellet

Relative Amounts of Phases										
Experimental Data		Major: Na ₃ (U,Pu)O ₄			Minor 25 wt % PuO ₂ -UO ₂ Solid Solution			Questionable Na ₁₁ U ₅ O ₁₆		
d Å	I/I ₁	d Å	I/I ₁ **	hk1	d Å	I/I ₁	hk1	d Å	i/i	hk1
3.40*	6							3.37	< 5	220
3.15	33				3.14	100	111	3.18	< 5	221
2.96*	10							3.01	25	310
2.787	77	2.777	77	111				2.755	76	222
2.730	26				2.72	48	200			
2.574*	8							2.549	22	321
2.417	100	2.405	100	200				2.386	64	400
2.240*	12							2.253	16	330
? 2.160*	6							2.190	19	331
? 2.024*	6							2.035	< 5	332
1.930	32				1.93	49	220			
1.880*	10							1.864	22	510
1.765*	12							1.780	19	520
1.733	68	1.700	68	220						
1.674	35				1.64	47	311	1.637	11	530
1.573	12				1.57	13	222	1.569	20	610
1.454	61	1.450	61	311				1.425	—	622
1.391	44	1.388	44	222						
1.365	10				1.36	9	400	1.365	—	444
? 1.285*	8									
1.255	15				1.25	18	331			
1.223	15				1.22	15	420			
1.205	37	1.202	37	400						
? 1.136	8									
1.124	15				1.11	13	422			
1.106	27	1.103	27	331						
1.087	35	1.075	35	420						
1.053	17				1.05	15	333			
? 1.017*	5									
0.984	36	0.982	36	422						

* Unidentified lines

? Questionable lines

** Intensities are those from experimental data

Na₃(Pu,U)O₄, face-centered cubic, A₀ = 4.81 Å ± 0.0225 wt % PuO₂-UO₂ solid solution phase, A₀ = 5.452 Å ± 0.009Na₁₁U₅O₁₆, cubic, A₀ = 9.543 Å

FIGURE 2. ARRHENIUS PLOT OF 300 HOUR THERMAL GRADIENT ANNEAL OF $U_{0.75}Pu_{0.25}O_{1.966}$ WITH 36.02 mg SODIUM

FIGURE 3. ARRHENIUS PLOT OF 100 HOUR THERMAL GRADIENT ANNEAL OF $\text{U}_{0.75}\text{Pu}_{0.25}\text{O}_{1.963}$ WITH 39.32 mg SODIUM

Previous tests in the presence of sodium exhibited bands of reaction product at the low temperature ends of the pellets in the cooler regions of the capsule. To verify that the observed band structure was due to the pellet interfaces, a 1-inch long pellet instead of the standard 1/4-inch long pellet was placed in the cold end of Capsule 6. Following the anneal, the long pellet had broken approximately one-third of the distance from the cold end. Reaction zones associated with interfaces were evident on both segments, and verified the band structure is indeed a pellet interface effect.

The capsules 1 through 7 contain solid solution fuel of 25% PuO_2 –75% UO_2 . Capsule 8 was loaded with $\text{PuO}_{1.8}$ pellets to determine the reaction tendency with plutonia. A large amount of sodium was added to allow for more oxygen movement within the capsule. Results will be reported next quarter.

It is premature to assess the results of capsules presented in Table 1, however, several points are emerging as significant:

1. The sodium-fuel reaction occurs in the cooler regions of the thermal gradient capsule.
2. The temperature of formation of reaction compound depends on the sodium temperature.
3. The temperature of the sodium apparently influences the threshold oxygen-to-metal ratio for formation of the reaction compound.

The last item may need further explanation since reaction compounds were observed in capsules 2 and 3 at 1.96–1.95 and in capsule 4 at 1.94–1.95. In capsule 4 the lowest temperature was 1110°C which results in an equilibrium sodium pressure of about 5 atm. In capsules 2 and 3 the lowest temperature was 900°C which corresponds to a sodium pressure of about 1 atm. The higher sodium pressure provides a driving force for stabilization of the reaction compound and causes it to form at a lower oxygen activity (or lower oxygen-to-metal ratio). The thermodynamic significance of this reasoning was under analysis at the end of the report period.

3. DETERMINATION OF COMPONENT ACTIVITIES

The determination of uranium activity in UO_2 – PuO_2 solid solutions is being studied by means of a transpiration technique with air and CO/CO_2 as the carrier gases. Experiments were conducted with UO_{2+x} and $(\text{U}_{0.6}\text{Pu}_{0.4})\text{O}_{2+x}$ at temperatures between 1300 and 1500°C with 10 CO_2/CO as the carrier gas. The results of these experiments are shown in Figure 4.

Good agreement was previously shown with Alexander's⁴ data for UO_{2+x} . The partial pressure of UO_3 over the mixed oxide is 20 to 40% less than that over UO_{2+x} of the same oxygen activity, an indication the stoichiometric solid solution of UO_2 –40% PuO_2 is ideal in behavior.

Experiments are in progress to characterize this solid solution in an air atmosphere for comparisons with the UO_2 – ThO_2 system⁵ and UO_2 – Y_2O_3 system.⁶ To verify the ideality of the $\text{U}_{1-y}\text{Pu}_y\text{O}_2$ solid solution for values of y other than 0.4, experiments are planned for $y = 0.2$ and $y = 0.6$ in the 10 CO_2/CO carrier gas.

4. OXYGEN MIGRATION IN $(\text{U,Pu})\text{O}_2$ SYSTEMS

The series of experiments described in the twelfth and thirteenth quarterly reports have been fabricated and are now undergoing or have completed thermal gradient anneals for periods from 100 to 2000 hours in the temperature range 1400–970°C. All the anneals will be completed during the next quarter.

The results of the 100-hour anneal containing $(\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_{1.933}$ are shown in Figure 5. The observed heat of transport of -2.36 kcal/mole is somewhat more negative than the -1.7 kcal/mole reported for similar material which underwent a 1000-hour anneal reported in the twelfth quarterly. The coldest eight pellets did not exhibit an appreciable stoichiometry gradient, an indication steady-state conditions had not been reached. The stoichiometry of these pellets, however, was higher than the over-all average for the capsule, an indication the pellets had picked up some oxygen during the 100-hour anneal. The pattern in evidence here is very similar to the behavior of $(\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_{1.97}$

FIGURE 4. UO₃ PARTIAL PRESSURE VS 1/T FOR UO_{2+x} AND U_{0.6}Pu_{0.4}O_{2+x}

FIGURE 5. ARRHENIUS PLOT OF 100 HOUR THERMAL GRADIENT ANNEAL OF $U_{1.75}Pu_{1.25}O_{1.933}$ -2.36 kcal/mole

in a similar temperature gradient as a function of time as reported in the ninth quarterly report. The only real difference is in the measured heat of transport and in the actual rates of approach to steady state. The apparent tendency in the behavior of these materials in a temperature gradient is to generate a highly negative heat of transport which slowly changes to a less negative steady-state value after long times. Further analysis of this phenomenon will be made when the four other capsules in this series are examined.

5. A THEORETICAL STUDY OF PLUTONIUM-URANIUM SEGREGATION IN MIXED OXIDE FUELS

Attention is focused on the vapor-phase evaporation condensation mechanism for Pu-O segregation.⁷ This mechanism is considered to be applicable to the restructured region of mixed oxide fuels which do not undergo center melting.

Computer calculations of the vapor pressures of urania and plutonia species over $U_{1-y}Pu_yO_{2-x}$, as functions of temperature and composition, are being performed with an ideal solid solution model. The various proposed values of the free energy of formation of gaseous⁸⁻¹⁰ PuO_2 are used as an additional variable. Comparisons between the calculated vapor pressures and recent effusion cell mass spectrometric vapor pressure measurements⁹⁻¹¹ show some rather serious discrepancies, principally between the measured and calculated oxygen pressures. Although it is not possible at this stage to identify the exact source of error, agreement between the calculated and measured vapor pressures of uranium-bearing and plutonium-bearing species over hypostoichiometric $U_{1-y}Pu_yO_{2-x}$ ($y = 0.15 - 0.20$) at about 2000°C is sufficiently good for use to have confidence the calculational method will reliably predict trends in uranium-plutonium segregations as the fuel oxygen-to-metal ratio changes at a given over-all value of y .

To this end, a series of steady-state radial temperature and oxygen-to-metal ratio profiles, covering the expected range of center temperatures and over-all oxygen-to-metal ratio values in irradiated fast ceramic reactor fuel pins, have been calculated with existing computer programs.¹² Results of the vapor pressure calculations will be combined with these data to yield steady-state radial plutonium gradients in the solid (restructured) fuel for a range of fuel linear power ratings and over-all oxygen-to-metal ratios. As before,⁷ the condition for stationary state with respect to uranium and plutonium vapor species has been assumed to be $\text{gradient } (Pu/U)_{\text{vapor}} = 0$.

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