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Calculation of the Liquidus and Solidus in the Plutonium-Uranium-Zirconium System

by A. D. Pelton and L. Leibowitz

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August 1988 ANL-IFR-93

CALCULATION OF THE LIQUIDUS AND SOLIDUS IN THE PLUTONIUM-URANIUM-ZIRCONIUM SYSTEM*

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*This report is an update of a report with the same title by A. D. Pelton, ANL-IFR-12 (May 1985).

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CALCULATION OF THE LIQUIDUS AND SOLIDUS
IN THE PLUTONIUM-URANIUM-ZIRCONIUM SYSTEM

by

A. D. Pelton and L. Leibowitz

ABSTRACT

A computer-coupled thermodynamic/phase diagram analysis of the Pu-U-Zr system has been performed to calculate the ternary solidus and liquidus surfaces.

Available data on the three binary systems Pu-U, U-Zr, and Zr-Pu have been collected and critically evaluated. Mathematical expressions for the thermodynamic properties of the binary phases have been obtained from this analysis. Ternary thermodynamic properties have been estimated from these binary properties, and the ternary phase diagram has been calculated there from. Error limits on the binary and ternary solidus and liquidus curves have been estimated. Expressions for the activity coefficients of all three components as functions of compositions and temperature in both solid and liquid ternary solutions have also been computed.

All calculations were performed with programs of the F*A*C*T (Facility for the Analysis of Chemical Thermodynamics) computer system based in Montreal.

INTRODUCTION

The techniques involved in computer-coupled thermodynamic/phase diagram analysis have been well documented [1-5]. When applied to the computation of an unknown ternary phase diagram, these techniques involve the critical evaluation and analysis of all relevant phase diagram and thermodynamic data for the three binary sub-systems with a view toward obtaining mathematical expressions for the thermodynamic properties of all binary phases as functions of composition and temperature. Following this, interpolation techniques based upon solution models are used to estimate the thermodynamic properties of the ternary phases from the properties of the binary phases. The ternary phase diagram is then calculated from the estimated ternary Gibbs energy surfaces.

For the Pu-U-Zr system, a liquid solution and a body-centered-cubic (bcc) solid solution exist at all compositions within the ternary system.

Liquidus and solidus curves have been reported for all binary systems, but data on the ternary liquidus or solidus are scarce. No reliable activity or calorimetric data on the binary solutions are available, and so the analysis of these systems must be based solely upon the reported binary phase diagrams.

In solutions in which deviations from ideal behavior are not large, polynomial expansions of the excess Gibbs energy in terms of the molar fractions are commonly used. In a binary system with components A and B, the excess Gibbs energy is given by:

$$G^E = X_A X_B (a_0 + a_1 X_B + a_2 X_B^2 + \ldots) \qquad (1)$$

where X_A and X_B are the molar fractions and $a_0, a_1, a_3, \ldots$ are empirical coefficients to be determined. It should be noted that:

$$G^E = RT(X_A \ln\gamma_A + X_B \ln\gamma_B) \qquad (2)$$

where γ_A and γ_B are the activity coefficients. In the general case, G^E varies with T, but in the case of the Pu-U-Zr system the available data are insufficient to permit the temperature dependence to be determined. Thus, it is assumed that G^E (and hence all coefficients $a_0, a_1, a_2, \ldots$) is independent of temperature. This is equivalent to assuming zero excess entropy.

If all coefficients $a_0, a_1, a_2, \ldots$ are zero, then the solution is ideal. If only a_0 is nonzero, then the solution is "regular." If a_0 and a_1 are nonzero, then the solution is "sub-regular." In the present case, no more than three coefficients were ever required in any binary solution.

Melting Points, Enthalpies, and Gibbs Energies of Melting

The Gibbs energies of melting of all three alloy components are required in the present analysis.

Data for Pu were reviewed by Oetting et al. [6], who reported a melting point of 640±2°C and an enthalpy of melting of 675±25 cal/mol. Heat capacities of the solid and liquid are also known with reasonable accuracy. The resultant expression for the Gibbs energy of melting is:

$$\Delta G^o_{fusion}\ (Pu) = -786 + 11.768\ T - 1.6\ T \ln T \tag{3}$$

In this, and subsequent equations, the temperature, T, is in Kelvins and the Gibbs energy is in cal/mol.

For Zr, data are taken from the JANAF Tables [7]. The melting point is 1852±5°C and the enthalpy of fusion is 5000±7% cal/mol. (These error limits are probably optimistic.) Solid and liquid heat capacities are also given in Ref. [7]. The resultant Gibbs energy of melting is:

$$\Delta G^o_{fusion}(Zr) = 2308 + 16.474\ T + 5.550 \times 10^{-4}\ T^2 - 2.446\ T \ln T \tag{4}$$

The data for U were reviewed by Oetting et al. [6] who reported a melting point of 1132±3°C. Two determinations of the enthalpy of fusion of uranium [8,9] were discussed [6], and the more-recent calorimetric value [8], 2185 cal/mol, was selected. However, there is a third experimental value [10] of 2.9 kcal/mol, as well as estimates of 3.25 kcal/mol [11] from vapor pressure data and 2.5 kcal/mol [12] from an assessment of uranium alloy phase diagrams. As is shown later, the value of 2185 cal/mol gives poor agreement with the measured Pu-U phase diagram. The Savage and Seibel [10] value of 2900 cal/mol, while not recommended by Oetting et al. [6], gives much better agreement with the Pu-U phase diagram. This presented us with a significant problem. On the one hand, two independent determinations of the Pu-U solidus were available. On the other hand, the recommended enthalpy of fusion of 2185 cal/mol seemed to be quite reliable. We resolved this dilemma in favor of the recommended enthalpy of fusion of uranium because it gave better agreement with our measurements reported below and it seemed unlikely that the experimental enthalpy measurement would be so much in error. Solid heat capacities were taken from Ref. [7] and the liquid heat capacity was taken from Ref. [6]. The equation used for the Gibbs energy of melting of uranium is:

$$\Delta G^{o}_{fusion(U)} = -1299 + 18.899\ T - 2.48\ T\ \ln T \qquad (5)$$

ANALYSIS OF BINARY SYSTEMS

The available literature on the binary systems was examined in this assessment including other reviews [13-18].

Pu-Zr System

The liquidus and solidus for Pu-Zr have been measured up to about 50 mol % Zr by Marples [19] and Bochvar [20]. These two studies disagree by as much as 50^{o}C in the liquidus and 100^{o}C in the solidus.

Another study by Taylor [21] for Zr concentrations up to 10 mol % gave a solidus close to that of Bochvar [20] but a liquidus 40^{o}C higher than that of either Marples [19] or Bochvar [20] at 10% Zr. In a fourth study, reported by the Mound Laboratory [22], liquidus temperatures 200^{o}C higher than those of all other authors were reported at Zr concentrations near 15 mol %. This study can probably be discounted.

Because the work of Marples [19] appears to be the most extensive, more weight was given to the results of this study in our analysis. This was also the opinion of Shunk [15] in his compilation.

Assuming ideal behavior for the liquid and regular solution behavior for the solid with

$$G^{E}(sol) = 1600\ X_{Pu}\ X_{Zr}. \tag{6}$$

we obtained the diagram shown in Fig. 1.

From this figure it can be seen that the calculated diagram is a compromise between the various reported diagrams, but gives more weight to the diagram of Marples [19].

The existence of small positive deviations in the solid phase is supported by the shape of the two-phase ϵ/δ region, which passes through a maximum at 640°C as can be seen in Fig. 1. Uncertainties are estimated to be ±50°C for the liquidus and ±100°C for the solidus.

Pu-U System

The liquidus and solidus for Pu-U have been measured by Ellinger et al. [23], and by Mound Laboratory [24].

The liquidus curves of these two studies agree to within better than 15°C, but the solidus curves diverge by up to 40°C. Ellinger et al. [23] state that they experienced difficulty in obtaining reproducible solidus measurements. They reported the minimum to be at 610°C and 12 mol % U on the basis of their solidus points. However, their liquidus measurements place the minimum closer to 620°C, which is the minimum temperature reported by Mound Laboratory [24]. Rosen et al. [25] report a minimum at 624°C and 9 mol % U.

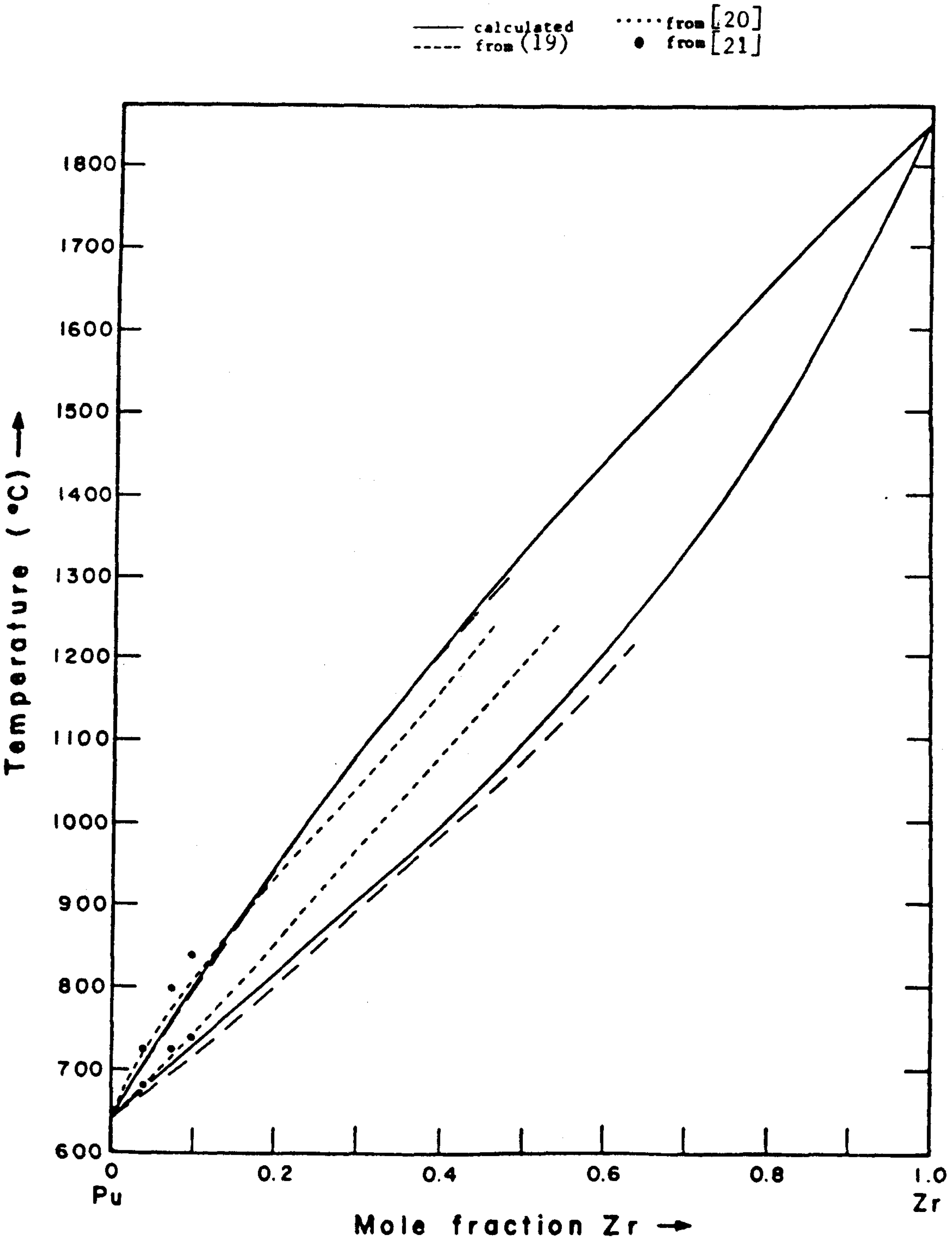

Fig. 1. Pu-Zr Phase Diagram

Activity measurements in the liquid phase were reviewed by Chiotti et al. [26]. Although the results suggest negative deviations in the liquid, they are so imprecise and their interpretation involves so many assumptions that they should be viewed cautiously. It is simpler, and also more concordant with experience in other alloy systems, to assume that the solidus/liquidus minimum is the result of small positive deviations in the solid. This contention is supported by the shape of the two-phase ϵ/η region, which passes through a maximum.

The liquidus in the Pu-U system seems to be relatively accurately known. Under the assumption that the liquidus is correct, it can be shown [27] that the solidus as well as $G^E(\mathrm{sol})$ can be exactly calculated if the enthalpies of fusion of the components as well as $G^E(\mathrm{liq})$ are known. We performed such calculations assuming ideal liquid behavior, using Eq. (3) for the enthalpy of fusion of Pu and taking the enthalpy of fusion of U to be 2185 cal/mol [6]. The resulting $G^E(\mathrm{sol})$ was small, sub-regular, and well behaved, but the calculated solidus was up to 25°C above that reported by Mound Laboratory [24] and 65°C above that reported by Ellinger et al. [23]. Reasonable agreement with the published solidus data was obtained, however, when the enthalpy of fusion of U was set at 2900 cal/mol, the value of Savage and Seibel [10]. Assuming a negative $G^E(\mathrm{liq})$ as suggested by Chiotti et al. [26] makes things worse. Fair agreement can be obtained if quite large positive excess Gibbs energies in both solid and liquid phases, which nearly compensate each other, are assumed. However, this seems improbable. It is more likely that either (i) the reported solidus curves are in error, or (ii) the enthalpy of fusion of U is higher than 2185 cal/mol. The former option seems more attractive in view of the experimental results reported below and of the precision of the enthalpy data of Stevens [8]. The excess Gibbs energy thus is given by

$$G^E(\mathrm{sol}) = X_{Pu}\, X_U (730 - 390\, X_U) \tag{7}$$

The resulting calculated phase diagram is shown in Fig. 2.

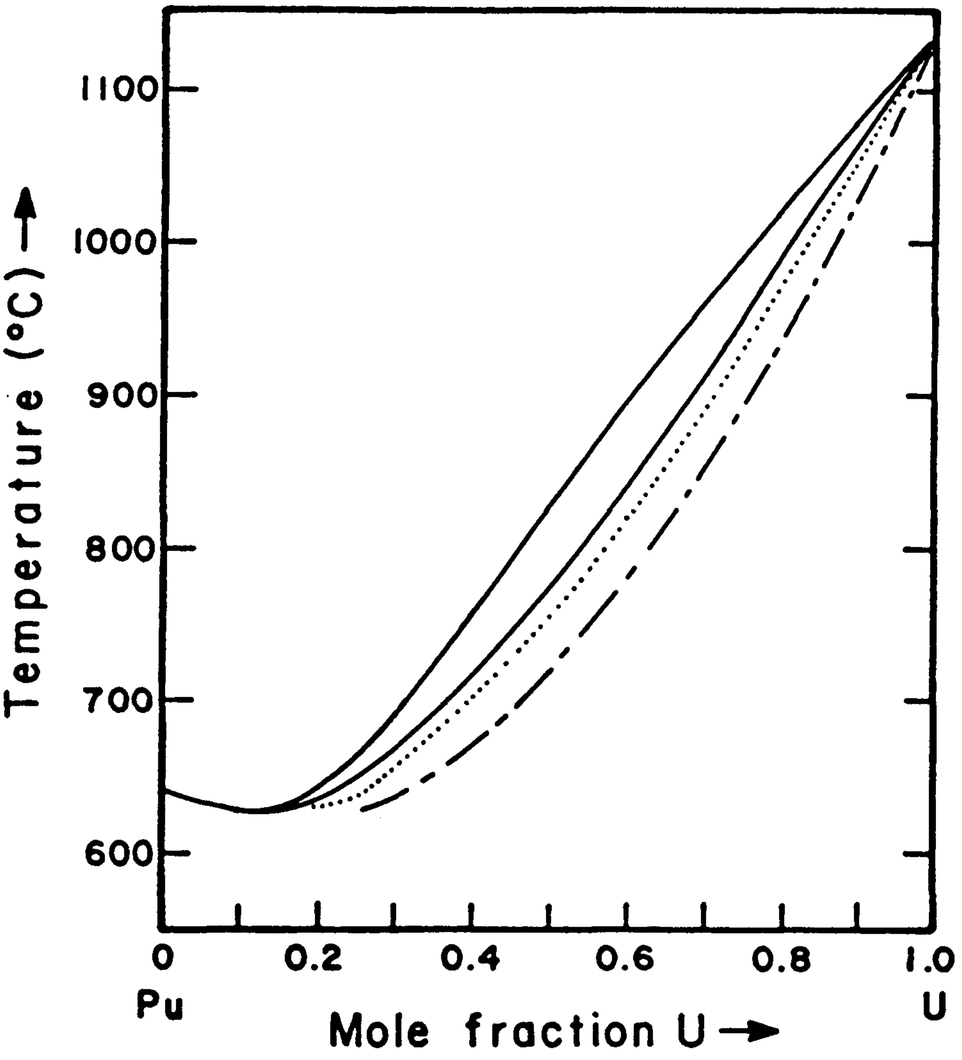

Fig. 2. Pu-U Phase Diagram

――――――――	Calculated
-------------	Ellinger [23]
....	Mound [24]

Because of the discrepancies discussed above, error limits are set at ±15°C for the liquidus and ±40°C for the solidus.

Zr-U System

The solidus and liquidus for Zr-U have been measured in only one study [28].

Another study [22] gives no data but reports that "solidus and liquidus temperatures are in agreement" with those of Summers-Smith [28] up to 50% Zr. The "lens-shaped" two-phase region shown in the Zr-U system is indicative of close to ideal behavior in both solid and liquid phases. However, the existence of a solid-solid miscibility gap at lower temperatures indicates quite strong positive deviations in the solid phase. Furthermore, the fact that the consolute composition of the gap is displaced toward higher U concentrations indicates that the positive deviations are quite asymmetric (nonregular). These positive deviations in the solid should then be expected to give rise to a minimum in the liquidus/solidus. That there is no such minimum can only be explained if the liquid phase also exhibits nonregular and nearly compensating positive deviations. Such behavior is unusual, and these observations thus add to the uncertainty surrounding the liquidus/solidus of this system.

Activity measurements in the solid solution as reviewed by Chiotti et al. [26] are so imprecise as to be of little value in the analysis.

More recent measurements [29] of the solid-solid miscibility gap place the consolute point at 772°C at X_U=0.70 with boundaries at the eutectoid temperature of 693°C at X_U=0.576 and X_U=0.89. These results are quite different from those of Summers-Smith, but are preferred by Shunk [15] in his compilation on the basis of better experimental technique. The following sub-regular equation was then calculated to reproduce the eutectoid compositions:

$$G^E_{(sol)} = X_{Zr}\, X_U\, (-688 + 5123\, X_U) \qquad (8)$$

Under the assumption of sub-regular behavior for the liquid, the following equation was computed in order to give a reasonable reproduction of the measured liquidus and solidus points:

$$G^E(\text{liq}) = X_{Zr}\, X_U\, (0 + 3800\, X_U) \tag{9}$$

The calculated diagram is shown in Fig. 3.

The S-shape of the solidus at higher U concentrations is a direct result of the positive deviations in the solid. The narrowness of the two-phase (liquid + solid) region at high Zr concentrations is a consequence of the enthalpy of fusion of Zr and must be reasonably correct.

This system is not at all well characterized. Error limits should probably be set at ±75°C for both the solidus and the liquidus.

CALCULATION OF THE TERNARY PHASE DIAGRAM

Excess Gibbs energies in the ternary liquid and solid phases were calculated from the values for the three binary systems by means of the "Kohler interpolation equations" [30]:

$$G^E = (1 - X_{Pu})^2\, G^E_{U/Zr} + (1 - X_U)^2 G^E_{Zr/Pu} + (1 - X_{Zr})^2 G^E_{U/Pu} \tag{10}$$

in which G^E at a ternary composition point is calculated from the values $G^E_{Zr/U}$, $G^E_{Zr/Pu}$, and $G^E_{U/Pu}$ in the three binary systems at the same values of the ratios X_U/X_{Zr}, X_{Zr}/X_{Pu}, and X_U/X_{Pu} as at the ternary point.

Calculated ternary isothermal sections are shown in Fig. 4.

Error limits for ternary compositions lying near a binary edge of the ternary composition triangle are approximately the same as the error limits

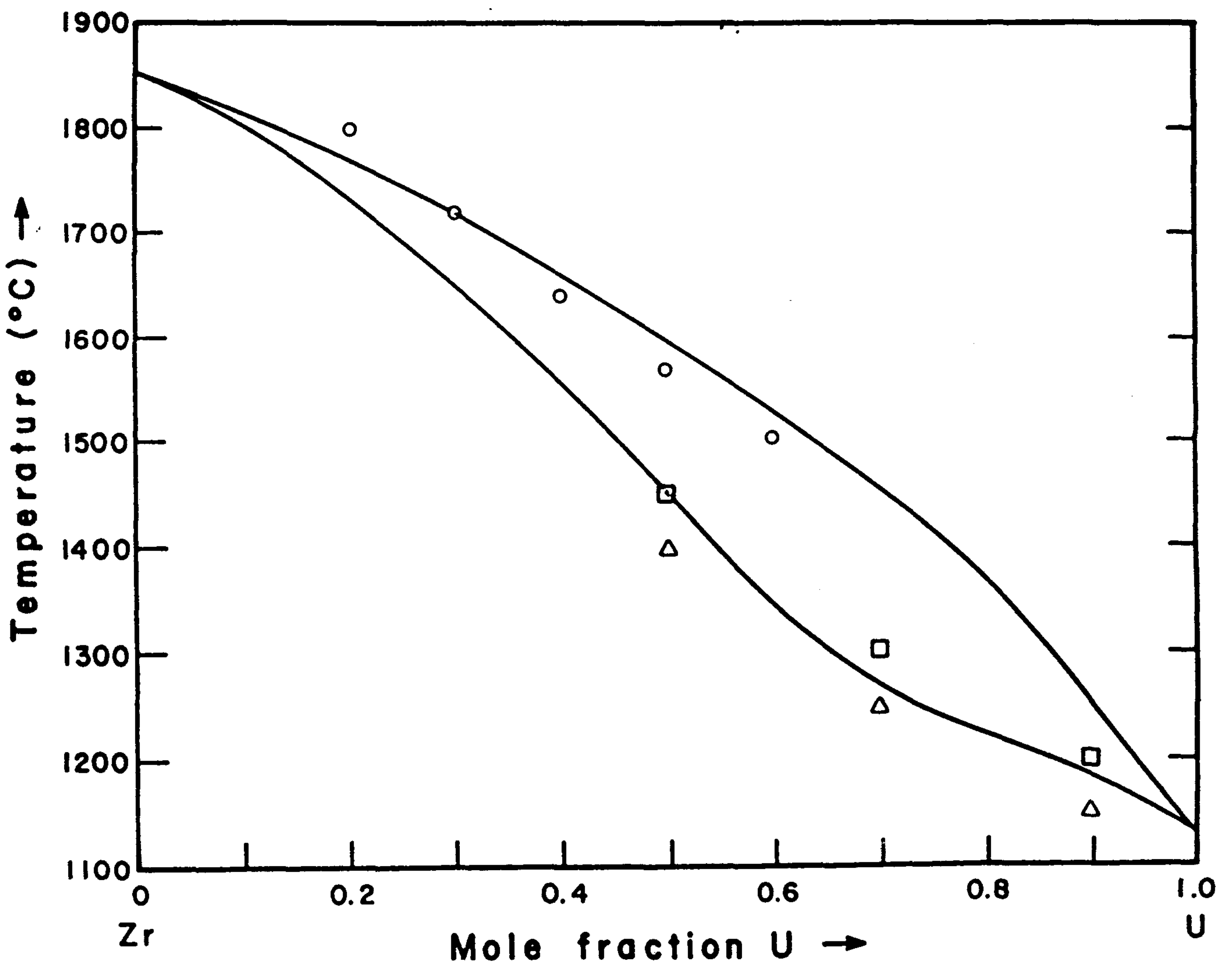

Fig. 3. Zr-U Phase Diagram

—— Calculated

Points from Summers-Smith [28]

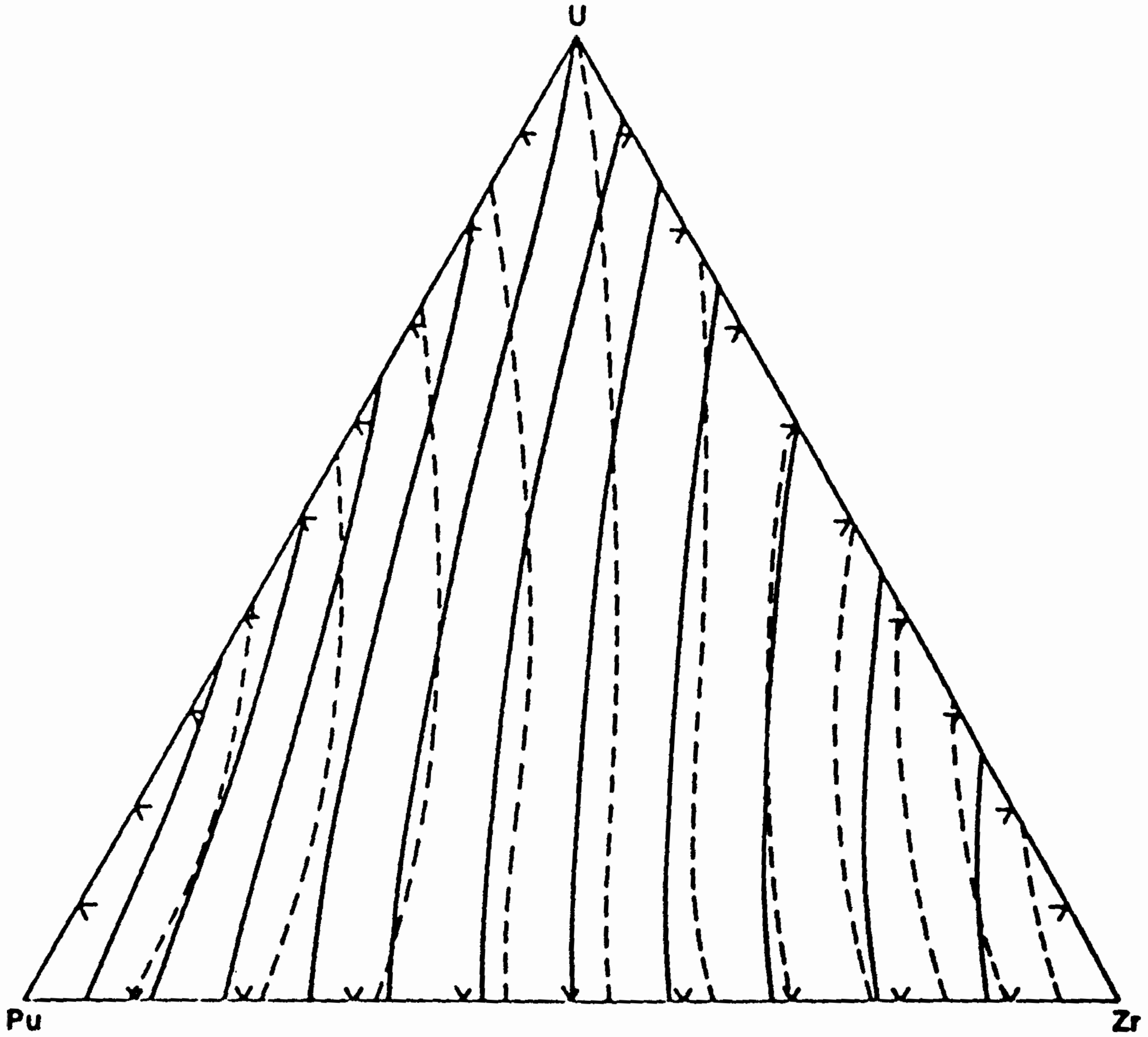

Fig. 4. Calculated Polythermal Projection of Liquidus (Solid Lines) and Solidus (Dashed Lines) for the U-Pu-Zr System. Temperatures Range from 700°C at the Corner to 1700°C at the Zr Corner in 100 °C Intervals.

at adjacent compositions in that binary, as given in the preceding section. For compositions near the center of the ternary composition triangle, an additional uncertainty due to the approximate nature of the Kohler interpolation technique must be included. Near the median of the composition triangle ($X_{Pu} = X_U = X_{Zr} = 1/3$), error limits are estimated as ±75°C for the liquidus and ±125°C for the solidus.

Measurements of the solidus and liquidus temperatures at two ternary compositions are reported by Harbur et al. [31]. The reasonable agreement of those results with our calculations is shown in Table I.

TABLE I. Solidus and Liquidus Temperatures of U-Pu-Zr Alloys [31]

Alloy(at.%)	Solidus(°C)	Liquidus(°C)
U-13 Pu-16 Zr	1105	1240
	1121(calc.)	1268(calc.)
U-12 Pu-29 Zr	1195	1425
	1187(calc.)	1394(calc.)

Recent measurements at Argonne [32], presented in Table II, show similarly reasonable agreement with the model predictions.

TABLE II. Solidus and Liquidus Temperatures of U-Pu-Zr Alloys [32]

Alloy(at.%)	Solidus(°C)	Liquidus(°C)
U-19.3 Zr	1216±7 1221(calc.)	1358±10 1371(calc.)
U-19.5 Pu-3.3 Zr	996±5 1012(calc.)	1050±4 1060(calc.)
U-19.3 Pu-14.5 Zr	1093±8 1071(calc.)	1321±23 1216(calc.)

In view of the difficulties encountered by all authors in measurements in the binary systems, the error limits on the experimental values above must be at least as great as the difference between the reported and calculated values.

Ternary isotherms in the Pu-corner of the diagram are reported by [22]. However, this study gives liquidus temperatures in the Pu-Zr binary which are 200°C higher near 15 mol % Zr than those reported by any other author. Hence, these results should be discounted. Nevertheless, the general shape of the reported isotherms is the same as that of the calculated isotherms.

Activity coefficients in both ternary phases can be calculated by differentiation of the Kohler equation. The calculated activity coefficients (referred to the pure bcc solids as standard states for the solid solution and to the pure liquids for the liquid solution) are determined as follows:

Let the components be numbered Pu = 1, U = 2, Zr = 3 with mole fractions X_1, X_2, X_3. In the following expressions, $R = 1.987$.

Liquid Solution

$$RT\ln_{\gamma 1} = -3800\, X_2^2\, X_3\, /(1 - X_1) \qquad (11)$$

$$RT\ln_{\gamma 2} = 3800\, X_2X_3\, (X_1X_2 + 2X_3)(1 - X_1)^2 \qquad (12)$$

$$RT\ln_{\gamma 3} = 3800\, X_2^2\, (X_2 - X_3 + X_1X_3)/(1 - X_1)^2 \qquad (13)$$

Solid Solution

The following general equation may be applied:

$$RT\ln_{\gamma k} = -(1-X_k)^2 f_{ij} + (1 - X_i)(f_{k(k)j} + X_i f_{kj}) + (1 - X_j)(f_{k(ki)} + X_j f_{ki}) \qquad (14)$$

$$\begin{aligned} f_{13} &= 1600(1 - t_{13})t_{13} \\ f_{1(13)} &= 1600t_{13}^2 \qquad \text{where } t_{13} = X_3/(X_1 + X_3) \qquad (15) \\ f_{3(13)} &= 1600(1 - t_{13})^2 \end{aligned}$$

$$\begin{aligned} f_{23} &= (1 - t_{23})\ t_{23}\ (-688 + 5123t_{23}) \\ f_{2(23)} &= (1 - t_{23})^2(-688 + 10246t_{23}) \quad \text{where } t_{23} = X_2/(X_2 + X_3) \qquad (16) \\ f_{3(23)} &= -5811t_{23}^2 + 10246t_{23}^3 \end{aligned}$$

$$\begin{aligned} f_{12} &= (1 - t_{12})t_{12}(730 - 390t_{12}) \\ f_{1(12)} &= 1120t_{12}^2 - 780t_{12}^3 \qquad \text{where } t_{12} = X_2/(X_1 + X_2) \qquad (17) \\ f_{2(12)} &= (1 - t_{12})^2\ (730 - 780t_{12}) \end{aligned}$$

By setting one of the mole fractions equal to zero in Eqs. (11-17), activity coefficients in any of the three binary sub-systems can also be calculated. In view of the many approximations which have been made, computed values of ln γ should be considered accurate to within a factor of 2.

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