Oxygen – Plutonium – Uranium

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Introduction

The interest in the O-Pu-U system arises from the fact that these oxides are of great importance in fuel applications for power reactors. Mixed plutonium-uranium oxides containing about 13-30% PuO_2 have been used as fuels for fast reactors in many countries. Uranium-plutonium mixed oxides containing 40% and higher amounts of PuO_2 are being considered potential fuels for the plutonium burner reactors [2004Kan]. Consequently, the knowledge of thermodynamic properties for these materials is of great importance.
Experimental investigations and calculations have been performed on the O-Pu-U system. The system has also been thermodynamically assessed. All the experimental and calculated investigations are reported in Table 1. [2001Car] have carried out a comprehensive review of the thermophysical properties of UO_2 and MOX fuels.

Binary Systems

[1998Che] has modeled the O-U binary system after performing a critical assessment of the available literature. Experimental incoherency found was the solubility of O in liquid uranium affecting the shape of the liquidus curve as well as the extension of the miscibility gap. Moreover the model used for the solubility of the $UO_{2\pm x}$ phase did not correspond to the real structure and the model of diatomic gas was considered in the calculation of the phase diagram. More recently, a progress in the thermodynamic modeling was presented by [2002Che] based on recent experimental data concerning the miscibility gap and the solubility of oxygen in liquid uranium. The model for the description of the $UO_{2\pm x}$ was also improved. However differences have been noticed in a later assessment done by [2002Gue]. It concerns the temperature of the invariant $L_2 \rightleftharpoons UO_{2\pm x}$ + Gas at 1 atm, varying from 2427°C [2002Gue] to 2800°C [2002Che]. No agreement was also observed concerning the shape of the miscibility gap in the hyperstoichiometry field which may exist up to the vaporization or be closed before. Due to these uncertainties, [2004Che] recently re-assessed the phase diagram taking into account recent experimental data. [1998Che] have calculated two different phase diagrams taking into account the small and large solubilities of oxygen. The assessment with small solubility of oxygen is accepted here as it is in agreement with [1998Gue] and also due to the fact that other accepted phase diagrams [2004Yam] take this assessment into account. As pointed out by [2004Che], the calculation of phase equilibria is not affected as the limit conditions are respected.
After [Mas2], a Calphad assessment of the O-Pu binary was carried out by [2003Kin] using thermodynamic modeling for the phase behavior and thermodynamic properties. This last version of the phase diagram is accepted in this assessment.
The binary boundary system Pu-U has been modeled by [1991Lei] and [1999Kur] and compared to the first version of the phase diagram presented by [1958Eli]. The phase diagram of [1999Kur] agrees well with experimental data but the calculated liquidus curve is shifted lower. Since version of [1999Kur] was used in the calculation of the ternary diagrams [2004Yam], which are accepted in the present evaluation this binary system has been accepted from [1999Kur]. The accepted binary phase diagrams of the Pu-U and O-Pu are shown in Fig. 1 and 2.

Solid Phases

The crystallographic data for the phases present in the O-Pu-U system and their ranges of stability are summarized in Table 2. Several space groups have been proposed in the literature for the U_3O_8 phase: $P\bar{6}2m$, *Cmcm* and *C*222. The most probable is certainly the space group *Cmcm* determined from the largest number of reflections (85 instead of 11 and 39 measured in other works [V-C2]). The major oxides of the respective binary systems PuO_2 and UO_2 form a complete range of solid solutions [2004Yam] with the

fluorite structure in the whole composition range and the ionic radii of Pu^{4+} and U^{4+} are very similar in the fluorite structure. It was found that the lattice parameters of stoichiometric uranium dioxide doped with plutonium ions decreased linearly with increasing plutonium up to 20 at.% [1998Tsu].

Quasibinary Systems

[1967Lyo] first studied the quasibinary system PuO_2-UO_2 showing the existence of complete miscibility between the two intermediate compounds. No later experimental work is available in the literature. The UO_2 fuel melts at a higher temperature than PuO_2, with values for UO_2 ranging from 2730°C to 2876°C [2001Car]. [2001Car] noticed also a large variation of the literature data for the melting temperatures of PuO_2 ranging from 2238°C to 2445°C. [1967Lyo] measured a melting temperature of PuO_2 at about 2390 ± 20°C, lower than the calculated value given by [2003Kin] (2467°C) in agreement with the calculated one proposed in the Calphad calculation of [1997Zha]. Experimental investigations are needed in order to estimate more precisely the melting point of PuO_2 fuels.
Recently, [2004Yam] have also calculated the PuO_2-UO_2 quasibinary system. The resulting thermodynamic dataset reproduces experimental data in the best possible way. This is also internally consistent with other accepted phase diagrams. Therefore, the calculated phase diagrams are accepted here. PuO_2-UO_2 are completely miscible in the entire composition range. Accurate determination of heat capacity of PuO_2 experimentally is difficult at high temperatures and this caused problems in the exact location of solidus and liquidus lines. [2004Yam] have made the calculation assuming ideal behavior. It concluded that the deviation from ideality is small as previously suggested by [1992Bea, 2001Car]. This is shown in Fig. 3.

Isothermal Sections

Phase studies on (Pu,U) oxides were carried out from 25 to 800°C [1967Ack, 1968Sar, 1969Koi1] within the limits $UO_{1.88}$-U_3O_8 and PuO_2-$PuO_{1.5}$. It was suggested that PuO_2 and Pu_2O_3 above 400°C could form a continuous solid solution. This is in disagreement with the large difference observed on the crystal structure for the two intermediate compounds. Moreover the intermediate compounds $PuO_{1.51}$ and $PuO_{1.62}$ are not indicated on the partial isothermal sections. Consequently, further experimental investigations are needed in order to determine precisely the phase equilibria in this composition range. Recently, [2004Yam] have modeled the ternary O-Pu-U system after careful analysis of the crystallographic data and thermophysical quantities. Isothermal sections have been calculated at 500 and 1000°C. This includes the calculation of interaction parameters and takes the non-stoichiometry into account. The data were verified by calculation of the oxygen potential of the fluorite phase in the hyper stoichiometric region. These calculated isothermal sections are shown on Figs. 4 and 5. Slight modifications have been made to bring them to agreement with the accepted binary systems. The calculated isothermal section at 1000°C has been modified along the U-Pu binary edge and the shape of the homogeneity range for the $(Pu,U)O_2$ solution has been also changed. The three- and two-phase equilibria involving the UO_3 phase have been suppressed since at 1000°C this phase does not crystallize according to the O-U phase diagram. Consequently, changes have been done and the three phase equilibrium U_3O_8 + Gas + PuO_2 has been added.

Thermodynamics

Thermodynamic data for the mixed (Pu,U) oxides have been obtained [1967Ack]. Several experimental data on the heat capacity and enthalpy increaments of unranium-plutonium mixed oxides have been reported [1972Lei, 1973Aff, 1974Gib, 1992Bea]. There is general agreement between the data except the data of [1972Lei] which are higher than the others. Agreement is observed in the correlations proposed by [1982Fin, 2000Fin, 2001Car, 2004Kan, 2004Yam] for the enthalpy increment and heat capacity data for uranium-plutonium mixed oxides. The details are reported in Table 3. [2004Kan, 2001Car] concluded that the enthalpies of $(Pu,U)O_2$ solid solutions in the temperature range 25-1527°C obey to the Neumann-Kopp molar additivity rule.

Oxygen potentials as a function of temperature were calculated for the mixed uranium-plutonium oxide at various O/M ratios and plutonium contents. A comparison of the calculated oxygen potential data of [1984Kri] with the measurements of [1977Tet, 1981Woo, 1979Woo] indicated fair agreements.
Vapor pressures of the various gaseous species above $Pu_{1-x}U_xO_{2-y}$ have been calculated or measured [1970Bat, 1976Osh1, 1976Osh2, 1977Bab, 1976Tet, 1978Ohs, 1982Bab, 2001Vis].

Notes on Materials Properties and Applications

[1973Ola] have carried out oxygen diffusion studies in the hypostoichiometric $(Pu,U)O_{2-x}$ system. This is important for the mutual segregation of fissile materials. [1999Kut, 2000Kut] have studied the shrinkage behavior and sintering kinetics of UO_2 for varying amounts of PuO_2. Thermal conductivity of mixed oxides $(Pu,U)O_2$ has been extensively reviewed [2001Car, 1982Mar, 1992Phi, 2000Dur] and mainly concerned high Pu content (about 20% Pu) mixed oxides for fast breeder reactors. The thermoconductivity of fuels strongly depends on temperature, porosity, composition, burnup and deviation from stoichiometry. [2001Car] noticed that the thermal conductivity decreases with temperature up to 1727°C and then increases with temperature. Addition of PuO_2 to the fuel or increasing porosity reduces the thermal conductivity. Burnup and/or deviations from stoichiometry have similar effects but they are negligible above 1927°C. Magnetic susceptibility of $(Pu,U)O_2$ has been investigated by [2002Kol] who have determined the ordering and Curie temperatures.

Miscellaneous

The behavior of $(Pu,U)O_2$ in a steady state temperature gradient between the liquidus and the solidus was investigated [2005Kle, 2001Kle]. The UO_2 and PuO_2 concentrations at the liquid-solid interface are discontinuous [2001Kle]. A severe redistribution phenomena of oxygen is observed in the temperature gradient of hypostoichiometric mixed oxides $(Pu,U)O_{2-x}$ which is enabled by intrinsic defects in the anion sublattice. It was concluded that the oxygen concentration gradient is controlled by the oxygen vacancy flux in direction to higher temperatures [2005Kle]. This process is quantified by the heat of transport which ranges from –10 $kJ \cdot mol^{-1}$ at the central void and about –230 $kJ \cdot mol^{-1}$ near the fuel surface [2001Kle]. For low temperature fuels in the absence of fission products, the steady state profile of the O/M ratio in the solid is established by flow of H_2O or CO_2 in the gas phase balanced by solid state diffusion of oxygen in the opposite direction [1973Ola]. The influence of this process upon oxygen distribution depends on the diffusion coefficients, densities and flow areas of solid and gaseous phases. [1973Ola] showed analytically how these factors affect oxygen redistribution in a closed axial thermal gradient geometry.
After irradiation of UO_2 fuel with about 3% ^{235}U enrichment, [1993Kle] observed that approximately 0.25% PuO_2 were produced per percent burnup. PuO_2 goes into the $(U,Pu, fp)O_2$ (fp means fission product) solid solution which results in an additional lattice contraction of $\Delta a = -18.5$ fm per percent burnup. The post-irradiation state of U,Pu mixed oxides nuclear fuels at 2% burnup was simulated by mixing 25 inactive fission products elements [1969Koi2].
[2001Vis] studied the vaporization chemistry of hypo-stoichiometric $(Pu,U)O_2$. The oxygen coefficient for hyper-stoichiometric uranium-plutonium oxide was measured and a slow oxidation was observed during a three month storage [1989Tes].
[2004Gib] has synthesized small ternary oxide clusters of uranium and plutonium oxide in order to study plutonium chemistry.
[1965Far1, 1965Far2, 1965Far3, 1965Far4, 1966Bar1, 1966Bar2, 1966Far1, 1966Far2, 1967Far1, 1967Far2, 1968Far1, 1968Far2, 1968Far3, 1968Far4, 1968Far5, 1969Fac1, 1969Fac2, 1969Far, 1970Bar] undertook a review of mechanical properties, method of fabrication and irradiation influence on mixed plutonium-uranium mixed oxides.

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Table 1: Investigations of the O-Pu-U Phase Relations, Structures and Thermodynamics

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1958Eli]	Chemical analysis, heat treatment, thermal analysis, dilatometry, X ray diffraction, metallography	600-700°C for 300 h / Pu-U
[1967Ack]	X-ray diffraction, metallography examination, emf	up to 1000°C / 64 to 73 at.% O and 40 to 33 at.% Pu
[1967Lyo]	Standard gravimetric analyses, X ray fluorescence and X ray diffraction	2300 - 2900°C / 5 to 85 mol% PuO_2
[1968Sar]	Metallography, X-ray diffraction	up to 1600°C / PuO_{2-x}, $1.62 \le x \le 2.00$ $(Pu,U)O_2$
[1969Koi1]	Differential thermal analysis, X-ray diffraction	25 - 800°C/ $(Pu_yU_{1-y})O_{2-x}$, $0.17 \le y \le 0.40$, $1.92 \le x \le 2.00$

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1970Bat]	Mass-loss and mass-spectrometric Knudsen effusion	1632 - 2138°C / $U_{0.8}Pu_{0.2}O_{2-x}$
[1972Lei]	Calorimetry	2075 - 2732°C / $(Pu_{0.2}U_{0.8})O_{1.97}$
[1973Aff]	Pulse heating method	up to 2727°C/ $(Pu_{0.2}U_{0.8})O_{2\pm x}$
[1976Osh1]	Laser pulse heating	up to 4727°C / 20 mol% PuO_2
[1976Osh2]	Laser pulse heating	up to 6727°C / 20 mol% PuO_2
[1976Tet]	Transpiration method	1877 - 2177°C/ $(Pu_{0.2}U_{0.8})O_{2-x}$
[1977Bab]	Laser surface heating method	up to 4727°C/ liquid UO_2 and $(Pu_{0.2}U_{0.8})O_2$
[1977Tet]	Equilibration technique	1777°C /$(Pu_yU_{1-y})O_{2-x}$, $0.15 \le y \le 0.30$, $1.92 \le x \le 2.00$
[1978Ohs]	Laser surface heating method	up to 4727°C/ liquid UO_2 and $(Pu_{0.2}U_{0.8})O_2$
[1979Woo]	Thermogravimetric analysis, solid-electrolyte galvanic cell technique	800-1000°C / $Pu_{0.25}U_{0.75}O_{2\pm x}$
[1981Woo]	Mass spectrometry analyses, oxygen potential-composition measurements	1000-1200°C / $Pu_{0.1}U_{0.9}O_{2-x}$, $Pu_{0.4}U_{0.6}O_{2-x}$, PuO_{2-x}
[2004Kan]	Inverse drop calorimetry technique	727-1507°C / $(Pu_yU_{1-y})O_2$ (y = 0.21, 0.28 and 0.4)

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(εPu,γU)	*cI*2 *Im*$\bar{3}$*m* W		continuous solid solution which exists between 1135 and 459°C [1999Kur]
(εPu) 640 - 483		a = 363.8	pure Pu, 500°C, [1989Pet] dissolves 100 at.% U [1999Kur] and 5.6 at.% Th at 605 ± 10°C [1985Pet1]
(γU) 1135 - 776		a = 352.4	pure U, 805°C, [Mas2] dissolves 100 at.% Pu [1999Kur] and 1.5 at.% Th at 1100°C [1985Pet2]
(δ'Pu) 483 - 463	*tI*2 *I*4/*mmm* In	a = 333.9 c = 444.6	pure Pu, 477°C, [1989Pet] the solubility of U is nearly absent [1999Kur] exists up to 443°C along the Pu-U binary [1999Kur]
(δPu) 463 - 320	*cF*4 *Fm*$\bar{3}$*m* Cu	a = 463.70	pure Pu, 320°C, [1989Pet] the solubility of U is nearly absent [1999Kur]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(γPu) 320 - 215	*oF*8 *Fddd* γPu	*a* = 315.87 *b* = 576.82 *c* = 1016.2	pure Pu, 235°C, [1989Pet] dissolves 0.8 at.% U at 280°C [1989Pet]
(βPu) 215 - 125	*mC*34 *C*2/*m* βPu	*a* = 928.4 *b* = 1046.3 *c* = 785.9 β = 92.13°	pure Pu, 190°C, [1989Pet] dissolves 2.0 at.% U at 272°C [1999Kur]
(αPu) < 125	*mP*16 $P2_1/m$ αPu	*a* = 618.3 *b* = 482.2 *c* = 1096.3 β = 101.79°	pure Pu, 21°C, [1989Pet], the solubilities of U is nearly absent [1999Kur]
(βU) 776 - 668	*tP*30 $P4_2/mnm$ βU	*a* = 1075.9 *c* = 565.6	pure, 720°C, [1989Pet] dissolves 25.5 at.% Pu at 702°C [1999Kur] exists down to 567°C along the Pu-U binary [1999Kur]
(αU) < 668	*oC*4 *Cmcm* αU	*a* = 285.37 *b* = 586.95 *c* = 495.48	pure, at 25°C [1989Pet] dissolves 14.2 at.% Pu at 567°C [1999Kur]
$(Pu,U)O_2$	*cF*12 $Fm\bar{3}m$		
UO_2 < 2852	CaF_2	*a* = 547.0	from 62.7 to 66.7 at.% O [2002Gue] lattice parameter at 25°C from [V-C2]
PuO_2 < 2467		*a* = 539.5	62.8 to 66.7 at.% O [2003Kin] lattice parameters [1990Wri]
U_3O_8 < 1870	*oC*44 *Cmcm*	*a* = 706.9 *b* = 1144.5 *c* = 830.3	melting point from [2004Che] lattice parameters from [V-C2]
U_4O_9 < 1123.5	*cI*832 $I\bar{4}32$ or $I4_132$	*a* = 2177	lattice parameter from [1989Rag]
UO_3 < 667.88	*oF*128 *Fddd* UO_3	*a* = 981.8 *b* = 1993 *c* = 971.1	[2004Che] lattice parameters from [V-C2]
Pu_2O_3 < 2020	*hP*5 $P\bar{3}m1$ La_2O_3	*a* = 383.88 *c* = 595.94	lattice parameters from [1990Wri] existence of a ordered superstructure [1967Ack]
$PuO_{1.61}$ 1069 - 353	*cI*80 $Ia\bar{3}$ Mn_20_3	*a* = 1099.1	from 61.7 to 63 at.% O [1990Wri] considered stoichiometric PuO1.61 by [2003Kin]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
$PuO_{1.52}$ < 417	*cI*80 $Ia\bar{3}$ Mn_2O_3	a = 1104.5	at 60.3 at.% O [2003Kin] lattice parameter from [1990Wri]
η, PuU 702 - 272	*tP*52	a = 1057 c = 1076	2.12 to 73.4 at.% U at 25 at.% U [1969Lea]
ξ, PuU < 625	*t***	a = 1069.2 a = 1065.1	25.9 to 74.6 at.% U at 25°C, 35 at.% U [1969Lea] at 25°C, 70 at.% U [1969Lea] $c/a \cong 1$

Table 3: Thermodynamic Properties of Single Phases

Phase	Temperature Range [°C]	Property, per mole of atoms [kJ, mol, K]	Comments
$Pu_{0.21}U_{0.79}O_2$	25 - 1527	$H°_T - H°_{298} = 79.895 \cdot T + 39.05 \cdot 10^{-4} T^2 + 160 \cdot 10^4 T^{-1} - 29534$	[2004Kan]
$Pu_{0.28}U_{0.72}O_2$	25 - 1527	$H°_T - H°_{298} = 77.841 \cdot T + 56.06 \cdot 10^{-4} T^2 + 149.17 \cdot 10^4 T^{-1} - 28710$	[2004Kan]
$Pu_{0.4}U_{0.6}O_2$	25 - 1527	$H°_T - H°_{298} = 78.263 \cdot T + 58.81 \cdot 10^{-4} T^2 + 151.66 \cdot 10^4 T^{-1} - 28944$	[2004Kan]

Table 4: Investigations of the O-Pu-U Materials Properties

Reference	Method/Experimental Technique	Type of Property
[1992Bea]	X-ray diffraction, electrical conductivity measurements, microcalorimetry	20-2000°C / $(Pu_yU_{1-y})O_{2-x}$ with 30 at.% Pu
[1999Kut]	Dilatometry	Density variation
[2000Kut]	Dilatometry	Sintering behavior
[2000Dur]	Laser Flash Method	Thermal conductivity
[2002Kol]	Superconducting Quantum Interference Device	Magnetic properties

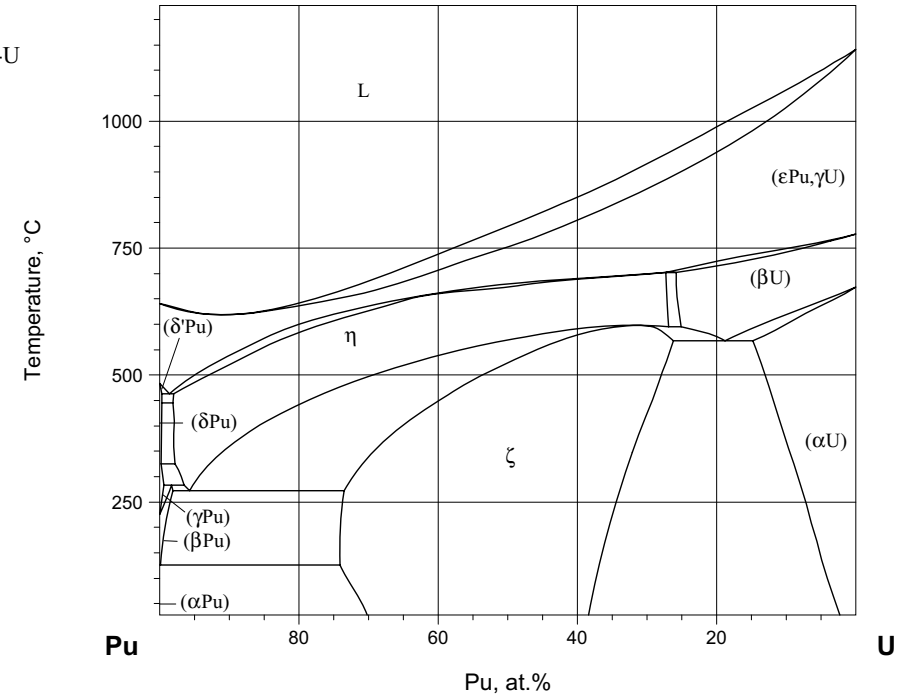

Fig. 1: O-Pu-U.
The calculated Pu-U phase diagram [1999Kur]

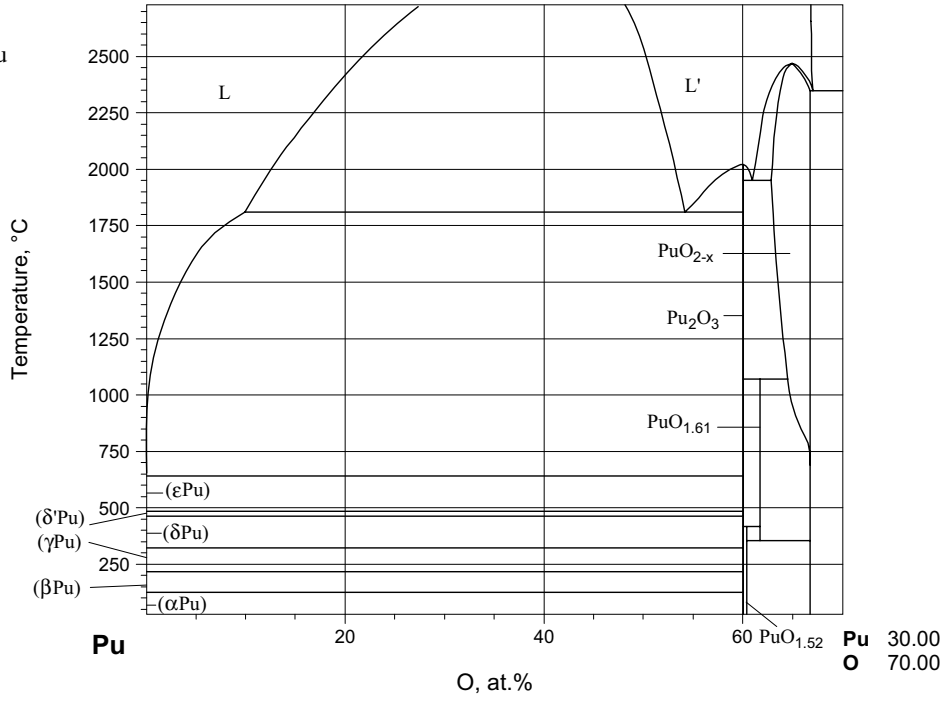

Fig. 2: O-Pu-U.
The calculated O-Pu phase diagram [2003Kin]

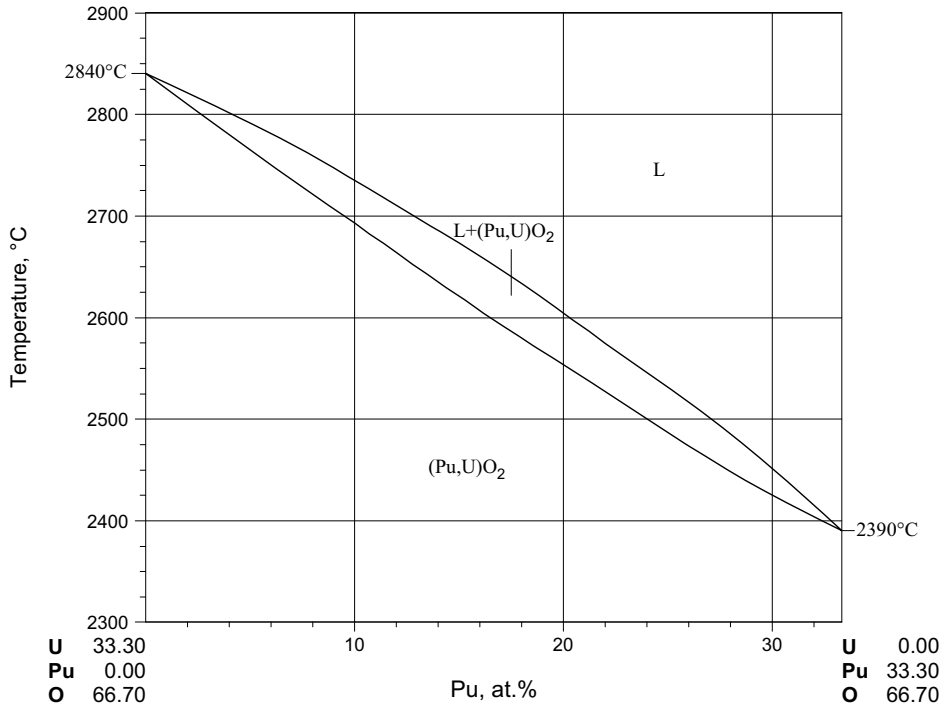

Fig. 3: O-Pu-U. The calculated PuO_2-UO_2 phase diagram

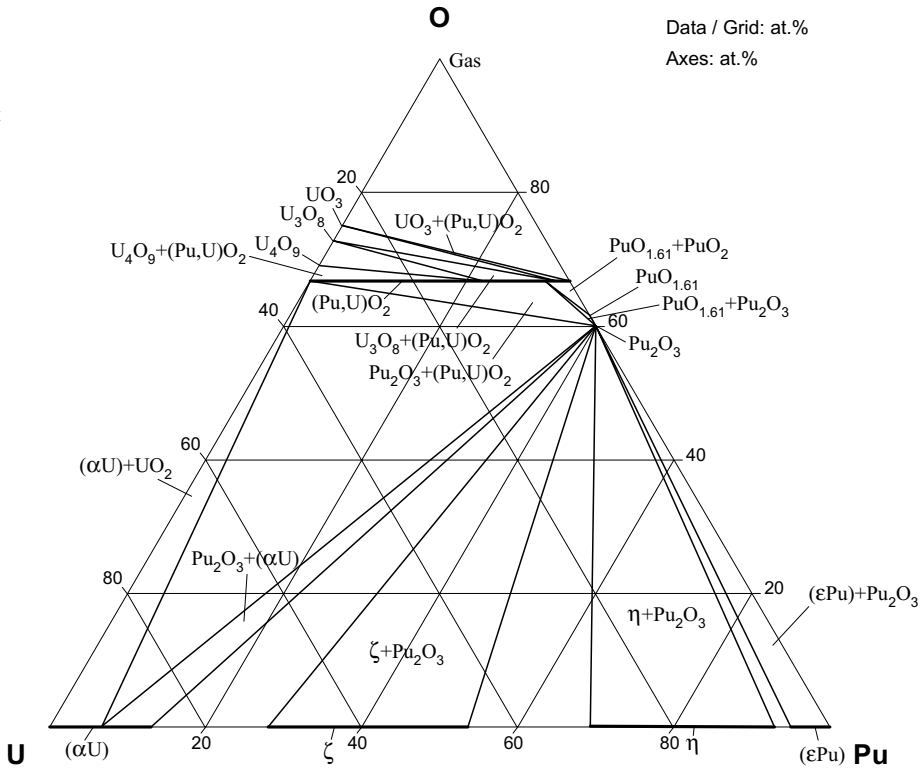

Fig. 4: O-Pu-U. The calculated isothermal section at 500°C

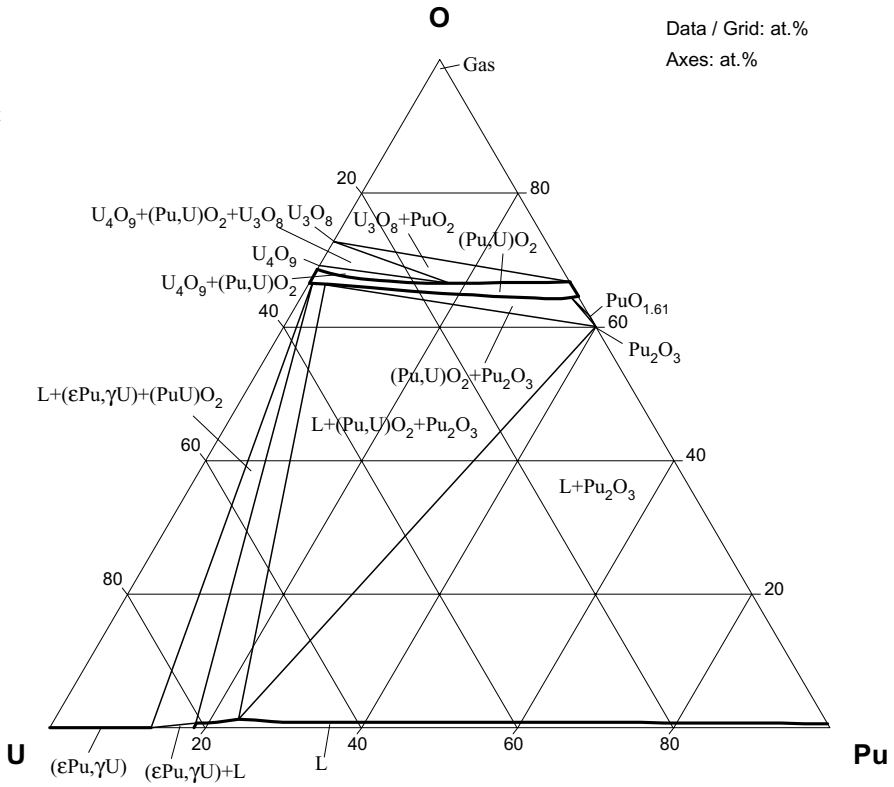

Fig. 5: O-Pu-U. The calculated isothermal section at 1000°C