Molybdenum – Oxygen – Uranium

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Introduction

The interest to this system arose mainly from the fact that Mo is one of most abundant products of fission. So it plays an important role in the chemistry of reactor fuel [1984Cha, 1997Nic]. The interaction of Mo with uranium oxides was studied by [1964Geb1, 1964Geb2, 1974Jen, 1993Kle, 2004Mar] experimentally and also by [1997Nic] from more theoretical point of view. [1974Jen] tested possibility to obtain UO_{2+x}-Mo composite materials by unidirectional growth from the melts.

Another point of interest to the system is solid state chemistry of ternary oxides containing both U and Mo. [1965Kov] studied section UO_2-MoO_2-MoO_3. 18 samples were annealed at various temperatures between 700 and 1000°C and studied by XRD. The results are presented in tabular form and as schematic section for 700 to 750°C. Three ternary phases were found and their crystal chemistry discussed basing on their X-ray characteristics (mainly lattice spaces); no detailed structure data were obtained. For the UMo_2O_8 phase a transformation at 1000°C was found by [1966Tru].

[1973Ser], [1974Ser1, 1974Ser2] performed analogous study of crystal chemistry (interrelation of structures through replacing U by Mo and formation of structural vacancies in the structure of βUMo_2O_8) of phases found in the section U_3O_8-MoO_2-MoO_3, though in this case no data on phase equilibria seem to exist.

Crystal structures and complicated crystal chemistry of various mixed oxide phases were studied also by [1990Sun, 1994Sun, 1995Tab, 1996Dya, 2004Kri]. These authors also studied mainly synthesis and structure of particular compounds, and virtually no data on phase equilibria are provided. For structural studies high resolution electron microscopy (HREM) was widely used.

Two vertical sections in oxide part of the system are studied. [1967Efr] investigated by thermal analysis part of the section between UO_2 and MoO_3 (50 to 100 mol% of the latter, *i.e.* $UMoO_5$-MoO_3). Two intermediate phases were found, in agreement with [1965Kov, 1966Tru].

The section UO_3-MoO_3 was investigated by [1973Ust]. Samples were prepared from MoO_3 and U_3O_8 and heated to 600 to 1000°C on air. The presence of phase "UO_2MoO_4" (= $UMoO_6$) indicated oxidation of U, so results were referred to the aforementioned section.

The review of crystal structures and phase relations of double oxides of U and Mo may be found in [1975Kel].

[1984Cha] investigated partial isothermal section at 1000 K in the region MoO_2-UO_2-O. They used XRD as well as emf measurement of O activity as a tool for investigation of composition of U oxide phases. Six ternary oxide phases were found.

Much attention was paid to determination of thermodynamic properties of some ternary oxides. [1984Cha] used their emf data for determination of the Gibbs energy of formation of $UMoO_5$. Using phase diagram data they estimated $\Delta_f G$ at 1000 K also for $UMoO_6$, U_2MoO_8 and UMo_2O_8. [1985Tri] studied reaction of vaporization of $UMoO_6$ by transpiration method at 1110 to 1250 K and derived $\Delta_f G$ for that compound. [1986Dha] tried to do the same using thermogravimetric analysis for determining the beginning temperature of reversible reaction of $UMoO_6$ with CaO. [1987Swa] also performed emf investigation of O activity in the mixture of $UMoO_5$ and $UMoO_6$. Basing on data of [1985Tri] for the latter, they derived thermodynamic properties of the former phase at 503 to 854°C. [2000Das] measured enthalpy increments for $UMoO_6$ at 26 to 727°C using high temperature Calvet microcalorimeter. Comparing the data of various authors for $\Delta_f H$ of $UMoO_6$ against proven empirical estimates, they chose the data of [1985Tri] for calculation of full set of thermodynamic properties of $UMoO_6$ including, in addition to $\Delta_f H$ and $\Delta_f G$, C_p, entropy, reduced Gibbs energy and reduced enthalpy for 25.15 to 1227°C.

[1985Tri] used own thermodynamic data to estimate a possibility of formation of a number of mixed U oxides during storage of nuclear wastes in the presence of lime.

[2000Abr] mentioned αUMo_2O_8 amongst potential ferroelectrics which crystallize in the space group *P*3 and satisfy to other criteria of ferroelectricity.
[2002Kan] measured rate of oxidation of alloy U-10 mass% Mo on air at 200 to 500°C.
[2003Tay] did not found any catalytic effect of U/Mo mixed oxide for propane or propene oxidation.

Binary Systems

All the three binary systems are accepted from [Mas2].

Solid Phases

The solubility of Mo in uranium oxides is essentially zero in both solid and liquid state [1964Geb1, 1964Geb2, 1974Jen, 1993Kle]. The same seems to be true for the mutual solubility of U and Mo oxides.
The formation of at least 16 ternary phases (double oxides) is known. Unfortunately, rather few data for phase equilibria seem to exist. In particular, the range of thermal stability for most phases remains unknown. For the γUMo_2O_8 phase obtained under hydrothermal conditions at low temperature [2004Kri] it is difficult to judge whether it is truly stable and present in the phase diagram.
For $UMoO_5$ the structural type UVO_5 was widely accepted in early works (including review [1975Kel]), but [1996Dya] established for this phase own structural type.
The known solid phases are listed in Table 2.

Isothermal Sections

The "schematic isothermal section for 700-750°C", suggested by [1965Kov], as well as section at 727°C from [1984Cha], do not contain numerous phases, synthesized and investigated by [1990Sun, 1994Sun, 1995Tab, 1996Dya] at nearly the same temperature and therefore are not presented here.

Quasibinary Systems

Figure 1 presents a partial section $UMoO_5$-MoO_3 (part of section UO_2-MoO_3) from [1967Efr]. $UMoO_5$ decomposes peritectically to liquid and UO_2 at 1087°C. Two more ternary phases exist in the section in addition to $UMoO_5$, namely UMo_2O_8, formed by a peritectic reaction at 1040°C in a high temperature α modification, and $UMo_{10}O_{32}$. The αUMo_2O_8 transforms into the βUMo_2O_8 at 1000°C. The $UMo_{10}O_{32}$ phase forms by peritectic reaction at 830°C. The temperature of the eutectic $L \rightleftharpoons UMo_{10}O_{32} + MoO_3$ is 780°C; its composition is estimated to be close to 1.5 mol% UO_2. We accepted the change of composition from $UMo_{11}O_{35}$, as was suggested for that phase in [1967Efr], to $UMo_{10}O_{32}$ as was derived in structural works of the same group [1973Ser, 1974Ser1]).
Figure 2 presents the section MoO_3-UO_3 after [1973Ust]. The phase $UMoO_6$ forms by a peritectic reaction at 980°C. The eutectic reaction $L \rightleftharpoons MoO_3 + UMoO_6$ takes place at 740°C and about 14.6 mol% UO_3 (in the text of original work this value is claimed to be in mass%, but as seeing in the figure, this must be mol%).

Thermodynamics

Table 4 presents accepted values of the Gibbs energies of formation for $UMoO_5$, $UMoO_6$, U_2MoO_8 and βUMo_2O_8.
For the $UMoO_5$ phase results of [1984Cha] and [1987Swa], both obtained by emf, are in good agreement.
The data for $UMoO_6$ include measurement of vapor pressure of $(MoO_3)_3$ above mixture of this phase with U_3O_8 [1985Tri], estimation of Gibbs energy at 1000K from that of $UMoO_5$ and phase equilibria data [1987Swa] and enthalpy increments, measured by [2000Das]. These data exhibit good mutual agreement and are accepted here; the results of [1986Dha] differ significantly and were rejected. The equation for enthalpy increments *H(T) – H(*298.15*)* suggested by [2000Das] is given in Table 5; full table for 298.15 to 1500 K may be found in the original work.
The Gibbs energies of formation of U_2MoO_8 and βUMo_2O_8 at 1000 K are estimated by [1984Cha] from their data for $UMoO_5$ and phase equilibria.

Notes on Materials Properties and Applications

[1974Jen] tried to obtain eutectic composites UO_2 with several metals, including Mo, by unidirectional growth from melt. This failed for mixtures Mo+$UO_{2.21}$ as neither solubility nor even wetting was observed, but succeeded for Mo+$UO_{2.08}$.
[2002Kan] measured the rate of oxidation of Mo-U alloys on air.

Miscellaneous

[1985Tri] used their thermodynamic data for investigation of possibility of formation of double oxides of U and Mo during storing of nuclear wastes in presence of lime.
[2000Abr] predicted ferroelectric properties for αUMo_2O_8.
[1997Nic] and [2004Mar] studied the state of Mo in UO_2 fuels.

References

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

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1964Geb1, 1964Geb2]	XRD, metallography	800 to 2600°C, section Mo-UO_{2+x}
[1965Kov]	XRD	700 to 1000°C, UO_2-MoO_2-MoO_3
[1966Tru]	XRD, lattice spacing	αUMo_2O_8
[1967Efr]	Thermal analysis	Section UO_2-MoO_3 (50 to 100 mol% of the latter)
[1973Ser]	XRD	$UMo_{10}O_{32}$ and modifications of $U_3Mo_{20}O_{64}$
[1973Ust]	Thermal analysis, XRD	Section UO_3-MoO_3 (samples prepared from MoO_3 and U_3O_8 and heat treated on air)
[1974Jen]	Unidirectional growth from melt	$UO_{2.14}$-5 mass% Mo and $UO_{2.07}$-7 mass% Mo
[1974Ser1]	XRD	$UMo_{10}O_{32}$ and modifications of $U_3Mo_{20}O_{64}$
[1974Ser2]	XRD, crystal structure	αUMo_2O_8
[1984Cha]	XRD, emf	The region MoO_2-UO_2-O, 727°C
[1985Tri]	Transpiration	$UMoO_6$, 827 to 977°C
[1986Dha]	Thermogravimetry, XRD	Temperature of beginning of reaction of $UMoO_6$ with CaO for thermodynamic properties of the former
[1987Swa]	Emf with ZrO_2/CaO electrolyte	Mixture $UMoO_6$ + $UMoO_5$, 503 to 854°C
[1990Sun]	HREM, XRD	UMo_5O_{16} phase
[1993Kle]	EPMA	Solubility of Mo in UO_2 at 900°C
[1994Sun]	HREM, XRD	Samples of UMo_5O_{17} and UMo_8O_{26} gross composition, 970K, anneal for 4 *d*
[1995Tab]	XRD, HREM	Mixtures of UO_2+MoO_2+MoO_3 1:1:1.25 (annealed at 800°C) and 3:2:18 (hydrothermal synthesis at 5400°C, 500 bar plus re-heating of product at 800°C)
[1996Dya]	XRD and HREM	$UMoO_5$, crystal structure
[2000Das]	Calvet calorimetry	$UMoO_6$, 26 to 727°C
[2004Kri]	XRD	$UMoO_6$, after hydrothermal synthesis at 220°C, 65 h

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(Mo) < 2623	*cI*2 $Im\bar{3}m$ W	*a* = 314.70	at 25°C [Mas2]
(γU) 1135 - 776	*cI*2 $Im\bar{3}m$ W	*a* = 352.4	[Mas2]
(βU) 776 - 668	*tP*30 $P4_2/mnm$ βU	*a* = 1075.9 *c* = 565.6	[Mas2]
(αU) < 668	*oC*4 *Cmcm* αU	*a* = 285.37 *b* = 586.95 *c* = 495.48	at 25°C [Mas2]
MoO_2 < 2300	*mP*12 $P2_1/c$ VO_2	*a* = 560.7 ± 0.1 *b* = 486.0 ± 0.1 *c* = 562.4 ± 0.1 β = 120.94°	[Mas2, V-C2]
Mo_4O_{11} < 818	*oP*60 $Pna2_1$ Mo_4O_{11}	*a* = 2449 *b* = 675.2 *c* = 545.7	[Mas2, V-C2]
Mo_8O_{23} < 818	*mP*62 *P*2/*c* Mo_8O_{23}	*a* = 1340 *b* = 404 *c* = 1680 β = 106.1°	[Mas2, V-C2]
Mo_9O_{26} < 815	*mC*280 *C*2/*c* Mo_9O_{26}	*a* = 2929.4 ± 0.3 *b* = 808.3 ± 0.1 *c* = 1681.6 ± 0.3 β = 95.47 ± 0.01°	[Mas2, V-C2]
MoO_3 < 810	*oP*16 *Pnma* MoO_3	*a* = 1385.5 *b* = 370.1 *c* = 396.2	[Mas2, V-C2]
UO_2	*cF*12 $Fm\bar{3}m$ CaF_2	*a* = 547.0	[Mas2, V-C2]
U_4O_9 < 1135	*cI*832 $I4_132$ U_4O_9	*a* = 2176	[Mas2, V-C2]
U_3O_8	*hP*11 $P\bar{6}2m$ U_3O_8	*a* = 681.2 ± 0.1 *c* = 414.2 ± 0.1	[Mas2, V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
UO_3	*tI*64 $I4_1/amd$ UO_3	a = 690.13 ± 0.05 c = 1997.54 ±0.18	[Mas2, V-C2]
* $UMoO_5$ < 1087	*oP*28 *Pbaa* $UMoO_5$	a = 1274.6 ± 0.1 b = 734.94 ± 0.07 c = 412.52 ± 0.02	[1965Kov, 1967Efr, 1996Dya]
* $UMoO_6$ < 980	*mP*32 *P*21/*c* $UMoO_6$	a = 720.0 b = 548.0 c = 1359 β = 104°36'	[1973Ust]
* αUMo_2O_8 1040 - 1000	*hP*99 *P*3 αUMo_2O_8	a = 1730 c = 614.5	[1967Efr, 1974Ser2][a)]
* βUMo_2O_8 < 1000	*oP*44 $P2_12_12$ βUMo_2O_8	a = 411.5 b = 732.7 c = 2008	[1967Efr, 1973Ser][a)]
* UMo_7O_{22}	*o**90	a = 1975 b = 1436 c = 410.4	[1965Kov]
* $UMo_{10}O_{32}$ < 830	*oC*344 *Cccm* $UMo_{10}O_{32}$	a = 1618 b = 1448 c = 1974	[1967Efr, 1973Ser, 1974Ser1]
* $\alpha U_3Mo_{20}O_{64}$	*oC*348 *Cccm* $\alpha U_3Mo_{20}O_{64}$	a = 824.6 b = 2876 c = 1978	[1973Ser, 1974Ser1]
* $\beta U_3Mo_{20}O_{64}$	*oP*87 *Pccm* $\beta U_3Mo_{20}O_{64}$	a = 411.9 b = 1438 c = 1976	[1973Ser, 1974Ser1]
* $\gamma U_{1.5}Mo_{10}O_{32}$	*oP*43.5 *Pbmm* $\gamma U_3Mo_{20}O_{64}$	a = 413.4 b = 1433 c = 987.3	[1973Ser, 1974Ser1]
* UMo_5O_{16} (mon.)	*mP*22 *P*2 UMo_5O_{16} (mon.)	a = 990.26 b = 718.23 c = 413.40 β = 90.20°	[1990Sun, 1994Sun]
* UMo_5O_{16} (orth.)	*oP*22 *P*222 UMo_5O_{16} (orth.)	a = 988.50 ± 0.07 b = 716.72 ± 0.07 c = 413.32 ± 0.03	[1994Sun] structure determined on sample obtained under hydrothermal conditions and re-heated at 800°C
* U_2MoO_8	*oP*44 $P2_12_12$ U_2MoO_8	a = 673.4 ± 0.6 b = 2324 ± 1 c = 411.5 ± 0.3	[1975Kel] superstructure to U_3O_8

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* $U_{0.5}Mo_4O_{13}$	*o**	*a* = 726.2 ± 0.3 *b* = 3223 ± 2 *c* = 402.9 ± 0.2	[1994Sun]
* $U_{0.5}Mo_5O_{16}$	-	-	structure is close to UMo_5O_{16} but with half of U positions vacant [1994Sun]
* $U_{0.75}Mo_5O_{16}$	*oP*21.75 *P*222	*a* = 990.00 ± 0.08 *b* = 718.89 ± 0.06 *c* = 410.74 ± 0.04	[1994Sun]
* $U_{1.5}Mo_{13}O_{42}$	*o**	*a* = 720 *b* = 5220 *c* = 400	[1994Sun]
* γUMo_2O_8	*oP*11 *Pbca* UW_2O_8	*a* = 1019.09 ± 0.07 *b* = 958.57 ± 0.07 *c* = 1427.41 ± 0.11	[2004Kri] synthesized at 220°C by hydrothermal method; probably metastable

[a] note: in [1967Efr] high-temperature modification is called β, in all other works α

Table 4: Thermodynamic Data of Reaction or Transformation

Reaction or Transformation	Temperature [°C]	Quantity, per mol of atoms [kJ, mol, K]	Comments
$U + Mo + 5/2O_2 \rightleftharpoons UMoO_5$	503 - 854	$\Delta G = -1816.9+0.3748T$	[1987Swa], emf
$U + Mo + 3O_2 \rightleftharpoons UMoO_6$	298.15	$\Delta H = -1975.2$ $\Delta G = -1824.0$	[1985Tri, 2000Das] tabulated for 298.15 to 1500K in [2000Das]
$2U + Mo + 4O_2 \rightleftharpoons U_2MoO_8$	727	$\Delta G = -2433 \pm 8$	[1984Cha], estimated from data for $UMoO_5$, oxides of U and Mo and phase equilibria
$U + 2Mo + 4O_2 \rightleftharpoons$ βUMo_2O_8	727	$\Delta G = -1928 \pm 10$	[1984Cha], estimated from data for $UMoO_5$, oxides of U and Mo and phase equilibria

Table 5: Thermodynamic Properties of Single Phases

Phase	Temperature Range [K]	Property, per mole of atoms [J, mol, K]	Comments
$UMoO_6$	298.15 to 1500	$H(T)–H(298.15) = -53928.8 + 158.65T + 21.443\cdot10^{-3}T\,(K) + 14.077\cdot10^5/T$	[2000Das]

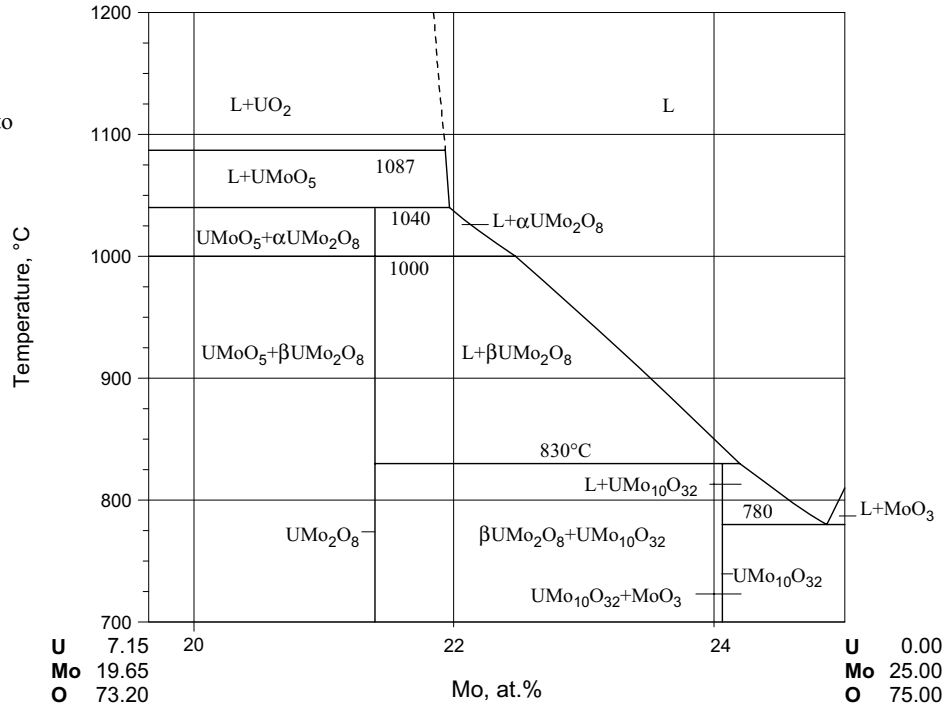

Fig. 1: Mo-O-U. The partial quasibinary section UO_2-MoO_3 for 50 to 100 mol% MoO_3 ($UMoO_5$-MoO_3)

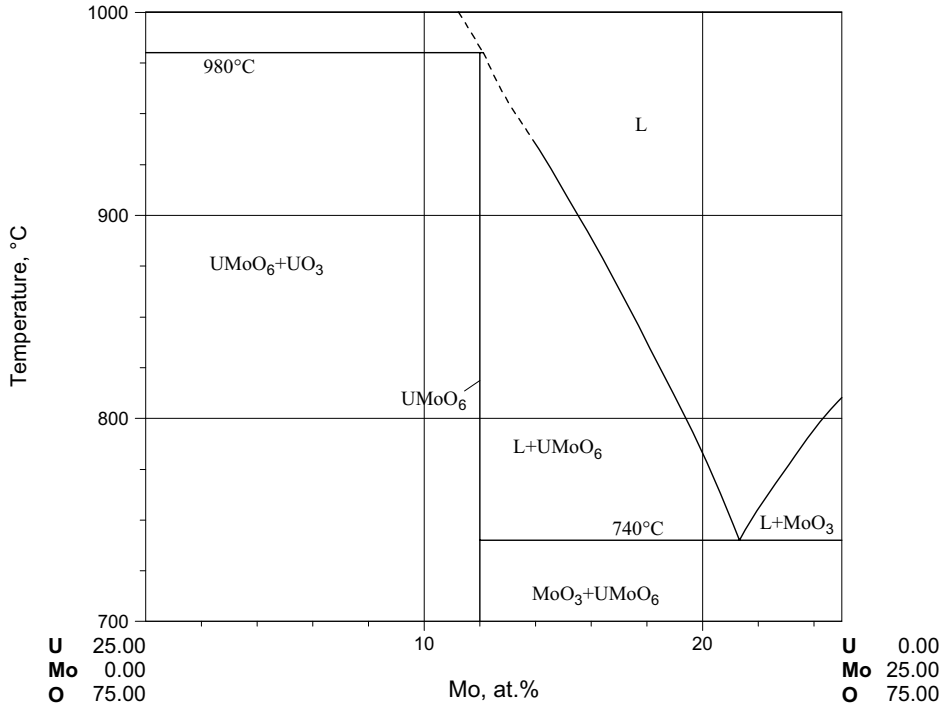

Fig. 2: Mo-O-U. The quasibinary section MoO_3-UO_3