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THE URANIUM-MOLYBDENUM BINARY SYSTEM

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Abstract

The uranium-molybdenum* system has been investigated by thermal, microscopic and X-ray methods. The system is characterized by a peritectic type of liquidus and solidus, extensive solubility of molybdenum in uranium, the lowering of the transformation temperatures of uranium and the absence of any intermetallic compound.

In view of the very sluggish solid transformations, phase boundaries in some regions have been drawn somewhat arbitrarily. Microscopic studies indicate one and two phase alloys in the general regions so indicated, but the exact limits of these transformations have not been determined.

The peritectic reaction occurs at 1285°C, and extends from 25 to approximately 95 atomic per cent molybdenum.

X-ray diffraction patterns of alloys in the gamma field show a body centered cubic structure, the lattice parameter varying with molybdenum content and giving by extrapolation, $a_0 = 3.467 \pm 0.005 \text{ \AA}$ for pure gamma uranium. The beta phase has also been retained by quenching low molybdenum alloys, its diffraction pattern indicating a complicated and as yet undetermined structure.

X-ray analyses have shown that the solubility of uranium in molybdenum is 1 atomic per cent at 900°C.

1. Metallographic Study of the Uranium-Molybdenum System

1.1 Introduction. Study of the uranium-molybdenum system was begun extensively with the discovery of the fact that small amounts of molybdenum in uranium gave the metal greatly increased hardness and in addition, heat-treatable alloys⁽¹⁾. Since the mechanism of this hardening was of prime interest, the most emphasis was placed on the study of uranium-rich alloys.

The first work on this system was carried out at "Y"⁽²⁾. In a series of heat treatments on a set of low molybdenum alloys (2 to 7 atomic per cent),

* Based on the reports: LA-42, LA-55, LA-68, LA-78, LA-120, CT-1497, CT-1501, CT-1515, CT-1775, CT-1779, CT-1780, CT-1784, CT-1977, CT-1982, CT-1985, CT-2374, CT-2483, CT-2616, CT-2632, CT-2700, CT-2778, and notebooks of D. H. Ahmann, A. S. Wilson, and A. I. Snow.

it was found that the gamma form of uranium could be retained by quenching, and to explain the microstructures and the hardening effect a constitutional diagram which included an eutectoid was postulated.

Thermal analyses carried out in this laboratory showed the solid solution areas to be different than predicted, but because of the sluggish transformations, the data could only indicate possible areas. To clarify this region, extensive heat treatments were carried out; from this work some areas were definitely established, but in higher molybdenum alloys the slowness of reaching equilibrium conditions made part of this region still doubtful. X-ray diffraction methods were used in the identification of phases and to establish the solubility limits in the gamma and alpha metal.

Dilatometric studies by the Battelle Memorial Institute ^(3,4) confirmed and more exactly defined the solid solution regions found by microscopic and thermal studies.

In this investigation little was done on the molybdenum-rich side of the system because of the difficulty in preparing these alloys, although the data indicate that the region is as postulated in the diagram.

Considerable work on the hardness and hardenability of uranium-molybdenum alloys has been done in other laboratories, including compression stress-strain studies and tensile strength tests on 2 and 5 wt. per cent (4.82 and 11.55 at.%) molybdenum alloys ^(2,5). In these studies, the greatest interest was in the 2 wt. per cent (4.82 at. %) molybdenum alloy, which appears to attain the greatest yield strength of any uranium alloy on soaking 4 hours at 700-900°C, quenching and reheating to 300-400°C ^(1,2). Also, some work on the thermal conductivity and other properties of these alloys was carried out ^(6,7,8).

1.2 Procedure. The alloys were prepared from "biscuit" uranium, the highest purity metal available from the Ames laboratory which contained 99.9+ per cent uranium, about 100 ppm C, 50-200 ppm Si, 30-100 ppm Fe, and 100 ppm Mg which was distilled during alloying. The molybdenum used was powdered metal obtained from the Fansteel Metallurgical Corp., analyzing 99.9 per cent molybdenum. This molybdenum powder was pressed into briquettes to facilitate handling. On melting, the uranium alloyed readily with the powdered molybdenum, and only short heating periods were required to produce homogeneous low-molybdenum melts.

The metals were alloyed by heating self-inductively to the desired temperature in a vacuum induction furnace, using zirconia or beryllia crucibles with either powdered graphite or beryllia as insulation. The alloys containing up to 70 at. per cent molybdenum were easily prepared at temperatures below 1500°C. By raising the temperature to 1700-1800°C satisfactory alloys containing up to 85 at. per cent molybdenum were formed; with higher molybdenum alloys, porosity and inhomogeneity were troublesome.

For thermal study a hole was drilled in the sample, and the borings were saved for chemical analysis. A chromel-alumel thermocouple, protected by a Mullite protection tube, was inserted into the hole and time-temperature curves were read and recorded by a Leeds-Northrup Micromax recorder. For these studies the cooling rate was from 2° to 8°C per minute.

The location of the solid solution and transition temperatures was also investigated by inverse rate thermal analyses using a Leeds-Northrup differential thermocouple setup. Since the samples tended to supercool and superheat, they were annealed above or below the transformation temperatures to complete the change and thereby aid in obtaining thermal arrests. This method met with limited success since, on heating, the transformations were so

gradual that the exact beginning or end of a transition could not be located. On cooling, the breaks were sharply defined, when they were not completely suppressed, probably because of supercooling. For this reason, the thermal data could only be used to fix the upper and lower limits of the transformation temperatures of a particular alloy. All the thermal results are listed in Table 1.

In the heat treatment experiments, the samples were annealed in a vacuum in a Leeds-Northrup annealing furnace in which the temperature was controlled to $\pm 3^{\circ}\text{C}$. An atmosphere of purified helium was introduced before quenching in water.

For microscopic study the samples were ground and polished by the usual metallographic techniques with a wet wheel polish of 600 mesh carborundum and a final electrolytic polish in a solution of 8 parts ethyl alcohol, 5 parts ethylene glycol, and 5 parts H_3PO_4 (85%). This electrolytic polish often brought out the microstructures although an electrolytic etch in 10 per cent oxalic acid was usually necessary for complete definition.

The results of dilatation studies by the Battelle Memorial Institute (3,4) are included in this report since they confirm and complete the findings of this laboratory in the low molybdenum solid solution region. Dilatation curves were run on 0.58, 1.87, 2.48, ca. 5 and ca. 10 wt. per cent (1.90, 4.51, 5.94, ca. 11.5, and ca. 21.6 at. per cent) molybdenum alloys. The samples were small cylinders 0.75 in. long and 0.5 in. in diameter machined from ingots melted and solidified in beryllia crucibles. Cooling curves were taken at rates from 1° to 3°C per minute and down to 0.5°C per minute, for the 2.48 wt. per cent (5.94 at. %) molybdenum alloy. The apparatus is similar to that used at the Bureau of Standards (9). Results of these dilatometric studies are shown graphically in Fig. 1.

TABLE 1

Molybdenum At.%	Chemical Analysis		Liquidus °C	Solidus °C	Transformations and Phase Changes
	Mo Wt.%	U Wt.%			
0			1133		770, 648
0.15	0.06	97.5	1133		770, 648
1.16	0.47	99.3	1130		714, 580
1.29	0.524		1132		721, 568
1.57	0.64	98.2	1135		683, 580
2.44	1.00	99.06	1120		688, 570 (683)
4.70	1.95	96.85	1140	1128	614 (644)
4.82	2				(622)
7.13	3				583
7.31	3.08	91.5	1150	1136	(590)
9.37	4				
11.6	5.02	95.14	1160	1123	(556)
12.1	5.25				532
12.8	5.56	89.76	1153	1137	
14.1	6.20	92.8	1171	1126	(588)*
15.7	7				
16.2	7.23	90.8	1193	1136	
17.3	7.76	89.1	1199	1142	
19.8	9.04	88.3	1232	1177	
22.2	10.33	88.0	1221	1150	
27.0	12.95				
30.4	15.00				
32.0	15.92	83.25	1300	1277, 1193	
38.5	20.16	78.5	1480	1285, 1227	1190
51.5	29.94	65.1	(none below 1500°)	1291	
59.5	37.2	59.4		1280	
70.8	49.5	44.8		1280	
81.4	63.9	33.94		1295	
93.3	84.7	15.13			

* Heating curve.

Temperatures in parenthesis were taken by differential thermocouples.

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SUMMARY OF PHASE CHANGES AS INDICATED BY DILATOMETER DATA

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The results of the chemical analyses are listed in Table 1. Considerable difficulty was found in the determination of uranium in the presence of molybdenum. A large number of analytical methods were studied, but in general were found to be unsatisfactory because of interference by the molybdenum ion⁽¹⁰⁾. The procedures found to be most successful were the distillation of molybdenum as molybdenum trioxide hydrochloride, $\text{MoO}_3 \cdot 2 \text{HCl}$,^(11, 12) or the precipitation of molybdenum as thorium molybdate, $\text{Th}(\text{MoO}_4)_2$ ⁽¹³⁾. After the removal of molybdenum, the uranium was determined volumetrically by reducing and then titrating with ceric sulfate. The precipitation method seemed to give the most exact analyses on alloys containing up to 70 at. per cent molybdenum. However, in some cases even this method accounted for only 97 per cent of the constituents in alloys whose microstructures showed no impurities. The alloys high in molybdenum were analyzed by precipitating uranium as the diuranate with ammonium hydroxide, and determining the uranium gravimetrically by igniting to the oxide, U_3O_8 .

1.3 Results. The uranium-molybdenum diagram as determined by this investigation is shown in Figure 2. The solidus and liquidus temperatures are based on the thermal data in Table 1, while the solid solution regions were determined from annealing, dilatometric and X-ray studies. The encircled points represent thermal data, while the crosses represent points determined by X-ray methods.

From zero per cent molybdenum, the liquidus rises slowly from 1131°C to 1285°C at 25 at. per cent molybdenum, then rises rapidly to the melting point of molybdenum, reported by Worthing as $2620 \pm 10^\circ\text{C}$ ⁽¹⁴⁾ and by other investigators as 2570°C ,⁽¹⁵⁾ and 2580°C ⁽¹⁶⁾. The solidus temperature

Figure 2. The equilibrium diagram of the uranium-molybdenum system.

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remains practically unchanged up to 15 at. per cent molybdenum, then increases to the peritectic temperature of 1285°C at 40 at. per cent molybdenum, and at approximately 95 at. per cent molybdenum rises rapidly to the melting point of molybdenum.

The gamma to beta transition of uranium is lowered by the addition of molybdenum, falling to 575°C at 7 at. per cent molybdenum. At this composition the beta phase disappears and the gamma changes directly to the alpha form of uranium. This transition temperature remains fairly constant to 17-18 at. per cent molybdenum and then appears to fall rapidly to 400°C at 22 at. per cent molybdenum. The sudden lowering of the transition temperature was predicted from microscopic studies, and was confirmed by X-ray study. In a 16.2 at. per cent molybdenum alloy annealed 2 weeks at 400°C , the two phases present were identified as alpha uranium, plus gamma uranium containing approximately 25 at. per cent molybdenum in solution.

The solubility of molybdenum in gamma uranium as determined by X-ray studies ranges from 35.8 at. per cent molybdenum at 1000°C to 29.9 at. per cent molybdenum at 700°C . The complete data are given in Table 3.

A number of annealing and quenching experiments were carried out to study the solid solution areas. In preliminary studies it was found that four hours was not sufficient to give equilibrium structures and that in alloys of molybdenum content greater than 8 at. per cent, 24 hours was not sufficient at temperatures below 650°C . In the first series, Fig. 3, a set of alloys, 0.15, 1.16 and 1.57 atomic per cent molybdenum, was quenched from 845, 735, 695, 640, 595 and 565°C , after annealing 24 hours. All the

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Temperature of Quench

845°C

735°C

695°C

640°C

595°C

565°C

0.15

1.16

1.57

Atomic per cent molybdenum

Fig. 3 Photomicrographs of low Mo content alloys annealed 24 hrs. X 250

779 011

samples at 845°C and the 1.57 per cent molybdenum sample at 735°C are in the one phase gamma region. The microstructures of the 0.13 and 1.16 per cent at 735°C and of all three samples at 695°C and 640°C seem to be in the same two phase region. One of these phases has been identified as beta uranium by the X-ray diffraction pattern of an alloy quenched in this region so the two phases are identified as gamma and beta uranium. At 600°C this two phase region has disappeared.

The second series, Figure 4, was run on alloys of compositions from 2 to 20 at. per cent molybdenum by quenching from 600, 500, and 400°C after a 24 hour anneal with additional quenches of 2 to 14 per cent molybdenum samples from 650° and 550°C , after annealing the same period of time. From these structures, the gamma plus beta region found in the first series can be extended to include the 2.44 per cent molybdenum sample at 700°C and 650°C and the 4.7 per cent molybdenum sample at 650° and 600°C . At 600°C in alloys containing more than 4.7 per cent molybdenum, areas appear to be eutectoid in nature. Precipitation along the crystal planes giving a Widmanstatten or Muntz metal structure can be seen in the 4.7 and 7.31 per cent alloys at 550, 500 and 400°C . The 11.6 per cent sample at 500° and 550°C shows a troctitic type structure while the alloys annealed at lower temperatures all show varying degrees of recrystallization but little else other than that they consist of two phases. These phases were identified as alpha and gamma uranium by X-ray analysis.

Figure 5 shows the microstructures of 7.31, 11.5, 13.8, 16.2 and 17.3 at. per cent molybdenum alloys annealed for 7 days at 400°C , and of 11.5 and 13.8 per cent molybdenum alloys annealed for 4 days at 500°C . Here equilibrium conditions are more nearly approached; in all the samples,

Temperature of Quench

700°C

650°C

600°C

550°C

500°C

400°C

2.44

4.70

7.31

11.60

Atomic Per cent Molybdenum

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Fig. 4 Photomicrographs of alloys (2 to 21% Mo) annealed 24 hours and quenched. X 250

13

12.8

14.1

16.2

17.3

20.9

Atomic Per cent Molybdenum

Fig. 4 (continued)

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Temperature of Quench

500°C

400°C

7.31

11.6

14.1

16.2

17.3

Atomic Per cent Molybdenum

Fig. 5 Photomicrographs of alloys (7 to 17% Mo) annealed 100 hours at 500°C or 170 hours at 400°C and quenched. X 250

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from 7 through 17 per cent molybdenum, the same eutectoid structure is present.

A final series of anneals of 24 hours at 1000°C and 3 days each at 500° and 400°C was carried out on 5 samples from 22 to 30 at. per cent molybdenum to check the extent of the solubility of molybdenum in gamma uranium and the limit of the alpha plus gamma two phase area. The samples after homogenization at 1000°C were one phase and were still one phase after annealing at both 500°C and 400°C (see Fig. 6).

Photomicrographs of the furnace cooled alloys of the composition from 38 to 93 at. per cent molybdenum (Fig. 7) show increasing amounts of the delta phase which is uranium dissolved in molybdenum. The relative amounts of the two phases present indicate that the delta solid solution begins at approximately 95 at. per cent molybdenum at 1285°C. The gamma uranium matrix in uranium-rich alloys shows precipitation of molybdenum as would be expected because of the decreasing solubility of molybdenum at lower temperatures.

The sluggishness of the transformations made the shape of the dilatation curves quite dependent on the heating and cooling rates used. It was also noted that the boundaries of the region of stability of the various phases was shifted. Whenever nearly isothermal transformations were observed as in the gamma-beta, beta-alpha and alpha-beta transitions in a 0.58 wt. per cent (1.90 at. %) Mo sample, a certain amount of overheating or undercooling is suspected.

From these studies it was decided that Figure 1 gives tentative stability boundaries of the various phases. The plotted points are selected from the dilatometer data to give the best estimate of the boundaries,

1b

1000°C

514°C

395°C

22.2

27.0

30.4

Atomic Per cent Molybdenum

Fig. 6 Photomicrographs of alloy series containing from 22 to 30% Mo homogenized 24 hours at 1000°C and annealed at 514 or 395°C. X 250

38.5% Mo

47.9% Mo

59.5% Mo

70.8% Mo

81.4% Mo

93.3% Mo

Fig. 7 Photomicrographs of furnace cooled alloys (38 to 93% Mo). Composition is indicated below each photomicrograph. X 250

although the sluggishness of the transformations in these alloys makes it difficult to draw the boundaries with any precision from the data obtained. It is apparent, however, that the lower limit of the gamma field drops rapidly with increasing molybdenum content up to 3 wt. per cent (7.25 at. %) molybdenum. The lower limit of the beta field, on the other hand, is not appreciably affected by molybdenum content and apparently the beta field extends to only about 2 wt. per cent (4.82 at.%) molybdenum. Further studies at lower heating and cooling rates may be expected to shift the boundaries of the various phases in the transition zones.

1.4 Discussion of Results. No discrepancies were found in the liquidus-solidus temperatures as determined by thermal analyses although the termination of the solidus arrest in low molybdenum alloys was often difficult to locate exactly. No liquidus temperatures were located with certainty above 1285°C.

The solid solution region has been constructed from microscopic, dilatometric and X-ray data, but is considered tentative since it does not explain the eutectoid structure. Since in the thermal data taken there is evidence of super-cooling, the breaks were used only to indicate the minimum extent of the solid solutions. The limits of stability of the various phases as defined by dilatation curves agree with the results obtained by microscopic study and these two methods have been used to draw this region. In all but the very small solid solution areas, the phases have been identified by X-ray analysis. For example, in the alpha plus gamma region, the two phases of a 16.2 at. per cent alloy annealed 2 weeks at 400°C were identified as alpha uranium plus gamma uranium containing approximately 25 at. per cent molybdenum in solution.

The region from 16 to 30 at. per cent molybdenum was not easily interpreted at temperatures below 700°C because of the extreme difficulty in obtaining equilibrium conditions. The limits of the areas were estimated from microscopic and X-ray evidence on samples annealed in this region and were confirmed by extrapolation from X-ray data and from microscopic work done at higher temperatures or on alloys of lower molybdenum content where equilibrium conditions could be assured.

As was mentioned earlier, little work was done on the high molybdenum alloys. The solubility of uranium in molybdenum was determined only at 900°C. This, with the extrapolation of areas from the microscopic study, was used to predict the limit of solution.

2. X-Ray Section

2.1 X-Ray Structures of Gamma and Beta Uranium. In the preliminary investigation of this system it was apparent that relatively small amounts of molybdenum depressed the uranium transitions⁽¹⁷⁾. It appeared likely that the gamma phase was stabilized enough to permit it to be quenched to room temperature. Attempts in this direction were immediately successful.

The first X-ray diagrams known to be of gamma uranium were of a 11.5 at. per cent molybdenum alloy⁽¹⁸⁾. This sample was body centered cubic, $a_0 = 3.43 \text{ \AA}$. Assuming the volumes of uranium and molybdenum to be additive, a spacing of $3.48 \text{ \AA}$ for pure gamma uranium at room temperature was found.

A more reliable value of the spacing of gamma uranium was obtained by determining the spacing as a function of the molybdenum content in conjunction with the studies on the limit of solubility of molybdenum in gamma uranium; this value ($a_0 = 3.467 \pm 0.005 \text{ \AA}$) was found from the data of

Table 2 by the graphical extrapolation shown in Figure 8. The density of pure gamma uranium at room temperature is 18.89 ± 0.05 g/cc, which is slightly less than that of alpha uranium at the same temperature.

The successful retention of beta uranium by the quenching of a low molybdenum alloy was also accomplished. By quenching a 1.31 at. per cent molybdenum alloy from 690°C , workers at Battelle were able to obtain a clear X-ray picture of the beta structure⁽²⁰⁾. This same sample when annealed at 800°C gave the characteristic gamma pattern, and when annealed at 600°C the alpha pattern was obtained. The lattice and the structure of beta uranium are unknown, but observed X-ray maxima indicate that the structure is probably of a low symmetry.

2.2 The Solubility of Molybdenum in Gamma Uranium. In the investigation of the solubility of molybdenum in gamma uranium at various temperatures by X-ray methods, the variation of the spacing with increasing molybdenum content was found. This was done by determining the lattice constants of one phase molybdenum alloys of varying compositions. (Table 2).

Table 2

Gamma Uranium a_0 in $\text{\AA}$	3 a_0	Atomic per cent Molybdenum
3.412 ± 0.001	39.72	17.2
3.394 ± 0.001	39.09	22.8
3.385 ± 0.001	38.78	24.2
3.376 ± 0.001	38.38	29.4
3.365 ± 0.001	38.07	31.2
3.140 ± 0.001	30.96	100.0

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Figure 8. The parameter cubed of gamma uranium plotted against the molybdenum composition as suggested by Mehl.

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The spacings listed in Table 2 were calculated according to Cohen's extrapolation method as recommended by Jette and Foote⁽²¹⁾ from diffractions patterns taken on a symmetrical self-focusing back-reflecting powder camera of 5 cm radius using $\text{CuK } \alpha$ radiation. The samples were prepared by sealing filings in quartz capillaries which were cleansed of corroding gases by means of a zirconium "getter" heated to 1100°C for several hours. These samples then were annealed for 24 hours at $800 \pm 10^\circ\text{C}$ and quenched in water. As shown by earlier metallographic work the molybdenum was completely dissolved in the uranium.

The relationships in Table 2 were plotted according to Mehl's modification of Vegard's law⁽²²⁾, (see Fig. 8). From this graph it was then possible to determine the amount of molybdenum dissolved in gamma uranium from the lattice constant of the phase and as mentioned previously, to find by extrapolation the lattice constant of pure gamma uranium.

An alloy containing 38 atomic per cent molybdenum was used for the actual determination of solubility. Previous microscopic and X-ray examination had shown this sample to be two phases (gamma solution of molybdenum in uranium and delta solution of uranium in molybdenum) at temperatures up to 1100°C with the gamma phase being predominant. The samples, prepared as before, were annealed and water quenched. The heat treatment and the results are listed in Table 3 and are shown graphically in Figure 9.

2.3 Solubility of Molybdenum in Alpha Uranium. The solubility of molybdenum in alpha uranium was checked by the determination of the lattice constants of a 16 at. per cent molybdenum alloy annealed at 500°C for 4 days. The lattice constants obtained are compared to those of pure uranium in Table 4.

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Figure 9. The solubility of molybdenum in gamma uranium as determined by x-ray methods.

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TABLE 3

Annealing Temperature	Annealing Time	Lattice Constant a_0 in Å	Molybdenum in Solution at. %
$1000 \pm 10^\circ\text{C}$	24 hrs.	3.352 ± 0.002	35.8
$900 \pm 10^\circ\text{C}$	24 hrs.	3.358 ± 0.002	33.5
$800 \pm 10^\circ\text{C}$	24 hrs.	3.361 ± 0.002	32.5
$700 \pm 10^\circ\text{C}$	48 hrs.	3.370 ± 0.002	29.9

TABLE 4

Molybdenum Alloy	Pure Uranium (23)
$a_0 = 2.854 \pm 0.01 \text{ Å}$	$a_0 = 2.852 \text{ Å}$
$b_0 = 5.837 \pm 0.01 \text{ Å}$	$b_0 = 5.865 \text{ Å}$
$c_0 = 4.968 \pm 0.01 \text{ Å}$	$c_0 = 4.945 \text{ Å}$

The atomic volume in both cases was 82.5 cubic Å. For this reason no appreciable solubility of molybdenum in alpha uranium is considered likely.

In this work, a non-symmetrical, self-focusing camera of 10 cm. radius was used, with CuK α radiation.

2.4 Solubility of Uranium in Molybdenum. Work on the solubility of uranium in molybdenum was limited; the results of only one run are being given. The spacing of the molybdenum phase of a 81.4 at. per cent molybdenum alloy powder annealed at $900 \pm 10^\circ\text{C}$, was determined.

The lattice constant ($a_0 = 3.1429 \pm 0.0003 \text{ Å}$) for the molybdenum phase of the alloy was appreciably larger than the lattice constant ($a_0 = 3.1394 \pm 0.0007 \text{ Å}$) of pure molybdenum as determined in this laboratory. The lattice parameter of the molybdenum phase corresponds to a solubility of approximately 1 at. per cent of uranium in molybdenum (determined at 900°C) as seen from Fig. 8.

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