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Journal of Alloys and Compounds 226 (1994) 89–93

Journal of  
ALLOYS  
AND COMPOUNDS

## Shear-like transformation in dilute U–W alloys

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Received 3 February 1994; in final form 5 April 1994

### Abstract

The retention at room temperature of the  $\gamma$ -U and  $\beta$ -U structures containing 0.43 and 1.0 at.% W respectively is achieved by rapid cooling from high temperature. The metastable  $\beta$ -U structure subsequently undergoes an isothermal shear-like transformation into a strained variant of the  $\alpha$ -U structure

*Keywords:* Uranium; Tungsten; Rapid cooling; Martensitic transformations

### 1. Introduction

The occurrence of martensitic transformations has been observed in several uranium-rich alloy systems. The addition of various solutes to uranium enables one to obtain the isothermal martensitic transformation  $\beta \rightarrow \alpha$  [1–3] or the athermal transformation  $\gamma \rightarrow \alpha$  [4–6]. The final product is generally supersaturated with the solute and slightly distorted. A literature survey revealed a lack of information on the uranium-rich side of the binary U–W phase diagram. Previous investigations [7–9] were concerned with the determination of the general features of the phase diagram and reported a small solubility of tungsten in  $\gamma$ -U, but neither  $\gamma$ - nor  $\beta$ -U was retained at room temperature even in the quenched structure [8,9]. The limited information available convinced us to undertake a systematic study of the uranium-rich side of this system. In this paper we present the main findings on previously unreported features associated with the uranium-rich alloys. These features include the metastable retention at room temperature of the  $\gamma$  and  $\beta$  structures. The  $\beta$  structure decomposes by a complex, martensite-like  $\beta \rightarrow \alpha$  transformation with isothermal characteristics into a strained but undistorted variant of the  $\alpha$ -U structure.

### 2. Experimental procedure

A master uranium alloy containing 27.3 at.% W and pure uranium were used to prepare dilute uranium

alloys with 0.43 and 0.84 at.% W. A third alloy with higher tungsten concentration (1.93 at.% W) was prepared from pure uranium and tungsten foil. The alloys were prepared in an arc-melting furnace under an atmosphere of purified argon and the various ingots were remelted several times to yield alloys with reasonable homogeneity. The tungsten content and the impurities of each ingot were checked by chemical analysis. The total impurities were less than 500 ppm and the deviation in tungsten concentration was within  $\pm 0.01$  at.%. The alloys were homogenized in a vacuum furnace at 1040 °C for 55 h and subsequently sliced into discs 4 mm high from the middle of each one. Optical microscopy X-ray powder diffractometry, scanning electron microscopy and electron probe analysis were employed for the structural and morphological examinations. Since the room temperature lattice parameter of tungsten is not modified by uranium [9, 10], its diffraction lines were used as internal standard for obtaining calibrated line positions.

### 3. Results and discussion

#### *3.1. Retention of the $\gamma$ phase: $\gamma$ quenched of sample containing 1.93 at.% W*

Metallographic examination of the as-cast sample containing 1.93 at.% W revealed a featureless structure that reacted very faintly and only after partial oxidation to polarized light. No metallographic feature characteristic of  $\alpha$ -U was observed (Fig. 1). Microprobe analysis

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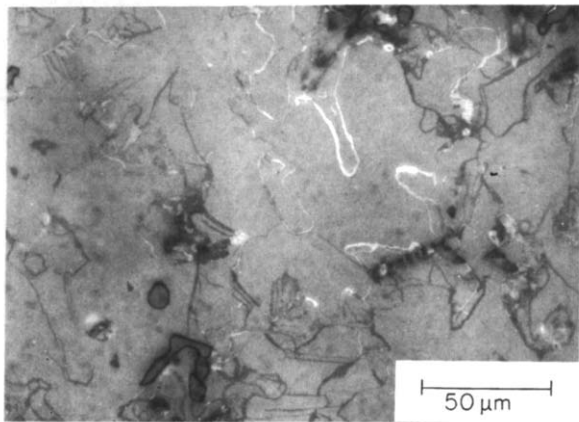


Fig. 1. As-cast U-1.93at.%W sample: an unidentified featureless structure with the absence of free tungsten particles in the matrix.

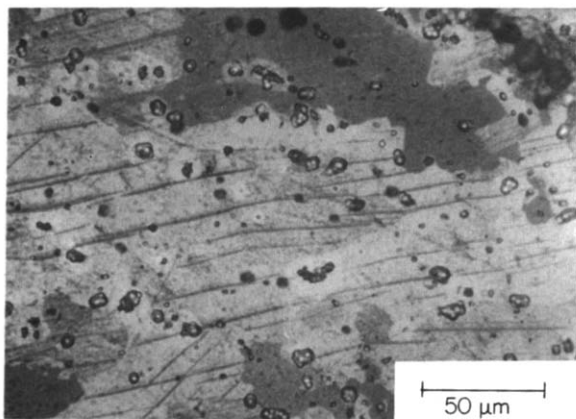


Fig. 2. U-1.93at.%W sample water quenched from 1060 °C: two different phases ( $\alpha + \gamma$ ) are clearly present with additional small precipitate particles identified as tungsten by microprobe analysis and X-ray diffraction.

showed a 1.8 at.% W content and X-ray diffraction of this sample yielded a combined pattern of  $\alpha$ -,  $\beta$ - and  $\gamma$ -U.

A sample containing 1.93 at.% W was annealed in a vacuum furnace at 1060 °C for 23 h and then water quenched. Metallographic examination of this sample revealed two different structures and small precipitates which were identified as tungsten particles (Fig. 2). X-ray diffraction analysis of this sample yielded a combined pattern of  $\alpha$ -U, a retained metastable  $\gamma$ -U and tungsten. The measured tungsten solubility in the matrix was between 0.8 and 1.0 at.%, which confirms the reported value [9] (about 0.9 at.% W at 1000 °C). The  $\alpha$  and  $\gamma$  lattice parameters, which were derived by least-squares fitting of the observed and calculated Bragg angles differences, are listed in Table 1. The observed and calculated data for the  $\alpha$  and  $\gamma$  phases are compared in Tables 2 and 3 respectively. The differences between the calculated and observed intensities are due to the presence of residual texture.

Table 1

Lattice constants of the three metastable allotropic uranium structures in dilute U-W alloys heat treated at high temperature and water quenched

Heat treatment	Structure	Lattice constants (nm×10)		
		a	b	c
Uranium unalloyed (room temperature values)	oC4 ( $\alpha$ )	2.8545	5.8681	4.9566
	tP30 ( $\beta$ )	10.59		5.6306
	cI2 ( $\gamma$ )	3.474		
U-0.43at.%W alloy heat treated at 930 °C for 30 min and water quenched	oC4	2.8563(54)	5.8686(11)	4.9571(9)
	tP30	10.594(1)		5.631(1)
U-1.93at.%W alloy heat treated at 1060 °C for 24 h and water quenched	oC4	2.8538(9)	5.8639(19)	4.9574(16)
	cI2	3.4665(5)		

Table 2

X-Ray diffraction data of cubic  $\alpha$ -U taken from water-quenched  $\gamma$ -U sample containing 1.93 at.% W

hkl	d (obs)	d (calc)	I (obs)	I (calc)	2 $\theta$ error
0 2 0	2.9343	2.9319	4.6	5.3	-0.025
1 1 0	2.5692	2.5660	100	70.5	-0.045
0 2 1	2.5248	2.5236	79.0	100	-0.018
0 0 2	2.4817	2.4787	52.2	51.3	-0.046
1 1 1	2.2797	2.2788	77.8	61.2	0.016
0 2 2	1.8924	1.8929	5.4	3.5	0.013
1 1 2	1.7826	1.7828	68.0	54.7	0.005
1 3 0	1.6115	1.6126	8.0	4.8	0.045
1 3 1	1.5334	1.5335	60.1	49.6	0.007
0 2 3	1.4390	1.4396	19.2	22.9	0.028
2 0 0	1.4267	1.4269	3.4	12.0	0.009
0 4 1	1.4065	1.4058	13.9	5.9	-0.039
1 1 3	1.3896	1.3893	16.3	16.6	-0.014
1 3 2	1.3515	1.3517	4.5	6.0	0.015
2 2 1	1.2417	1.2421	9.5	32.1	0.027
1 3 3	1.1542	1.1541	38.9	25.7	-0.010
1 5 0	1.0845	1.0848	18.6	13.6	0.028
2 4 0	1.0228	1.0225	22.3	9.9	-0.042

Table 3

X-Ray diffraction data of cubic  $\gamma$ -U taken from water-quenched  $\gamma$ -U sample containing 1.93 at.% W

hkl	d (obs)	d (calc)	I (obs)	I (calc)	2 $\theta$ error
1 1 0	2.4545	2.4505	55	100	-0.062
2 0 0	1.7345	1.7328	77	18.6	-0.048
2 2 0	1.2256	1.2253	17	13.1	-0.024
3 2 1	0.9261	0.9262	100	34.8	0.028

As seen in Table 1, the final data of the  $\alpha$ -U lattice parameters of the quenched sample are close to the pure uranium cell parameters within experimental error. However, in spite of the fact that there is no significant shift in the diffraction line positions, considerable line

broadening appeared in the  $\alpha$ -U diffractogram. From a broadening analysis using the Williamson–Hall method [11], it was found that internal microstrain is the main cause of the line broadening (Figs 3(a) and 3(b)). Independently we found that the microhardness of the  $\alpha$  grains was 363 DPN compared with 165 DPN in pure uranium. The increase in the hardness of the quenched alloy and the pronounced broadening of its

diffraction lines lead us to believe that the residual microstress is due to the metastable solubility of tungsten in the  $\alpha$ -U structure. Further confirmation of this statement is in progress and will be reported later.

The presence of retained high temperature  $\gamma$  phases in some uranium-based alloys, e.g. the binary uranium-rich U–Mo, U–Nb and U–Zr alloys, is well documented [12]. The tendency to form  $\gamma$ -like transition phases is limited to higher solute contents, in general much in excess of the  $\alpha$ -U solubility limit. The estimated parameter of the elementary cI2 cell at room temperature of 0.3474 nm for pure uranium [13] is larger than the value which was determined for the  $\gamma$ -U phase containing 1.0 at.% W with a contracted unit cell parameter down to  $a=0.34665$  nm (Table 1), which is close to the values reported for U–12at.%Mo and U–25at.%Nb [14]. It should be noted that in contrast with the U–Mo and U–Nb systems [14], the retention of the  $\gamma$  phase in the U–W system requires a lower atomic percentage, which affects its lattice parameter only slightly. From the results of this study tungsten was found as another  $\gamma$ -U stabilizer.

### 3.2. Martensite-like decomposition of the $\beta$ phase: $\gamma$ quench of samples containing 0.43 and 0.84 at.% W

Samples containing 0.43 and 0.84 at.% W were heat treated at 930 °C for 30 min and water quenched. During metallographic examination of the samples we observed the appearance of lenticular plates characteristic of a shear-like transformation (Figs. 4(a) and 4(b)).

A prolonged stay at room temperature led to thickening and lengthening of the existing plates and to the appearance of new ones. The sequence of micrographs in Fig. 5 shows the various stages of the development of the martensite plate as a function of the time elapsed since the samples had been quenched to room temperature.

Towards the end of the transformation (Fig. 6) the whole area under examination becomes a mosaic of ridges and depressions, the outcome of the upheaval that the polished surface has undergone. The pronounced surface relief is associated with the formation of  $\alpha$  phase plates by means of the shear-like transformation. The growth rate of the plates was measured in both directions and found to be  $0.3\text{--}1.5 \mu\text{m min}^{-1}$  in the longitudinal direction and  $0.03\text{--}0.08 \mu\text{m min}^{-1}$  in the transverse direction.

X-ray diffraction of the two samples immediately after quenching yielded a combined pattern of  $\alpha$ -U and  $\beta$ -U. A month later no traces of  $\beta$  were found in the X-ray diffraction patterns of the measured samples. The measured lattice constants of the various phases are given in Table 1. The occurrence of a  $\beta \rightarrow \alpha$  slow

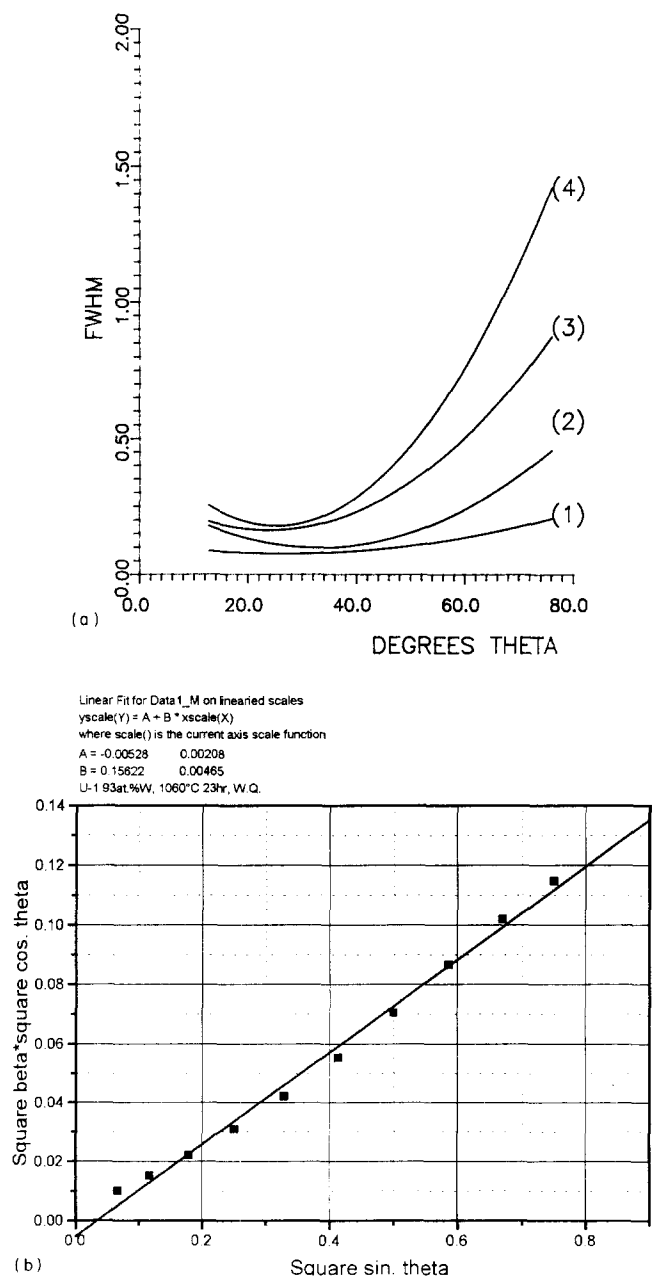
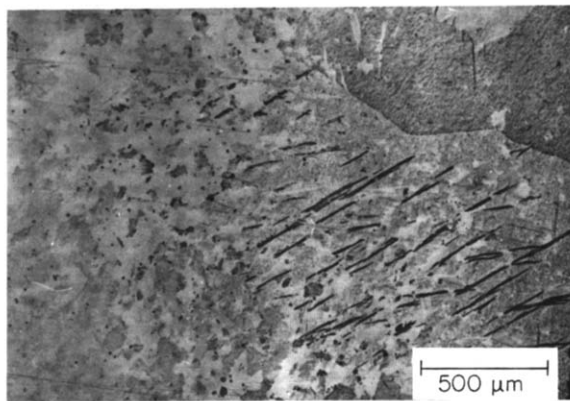
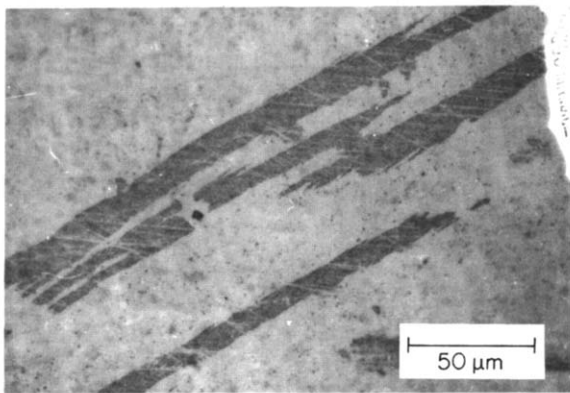


Fig. 3. (a) Comparison of FWHM (full width at half-maximum of profile) of measured reflection peaks of various uranium samples: (1) alumina flat plate, intensity standard (NIST-1976); (2) pure uranium furnace cooled; (3) U–0.43at.%W water quenched from 930 °C; (4) U–1.93at.%W water quenched from 1060 °C. (b) Interpretation of line broadening of sample (4) in which  $\beta \cos \theta$  is plotted against  $\sin \theta$ .



(a)



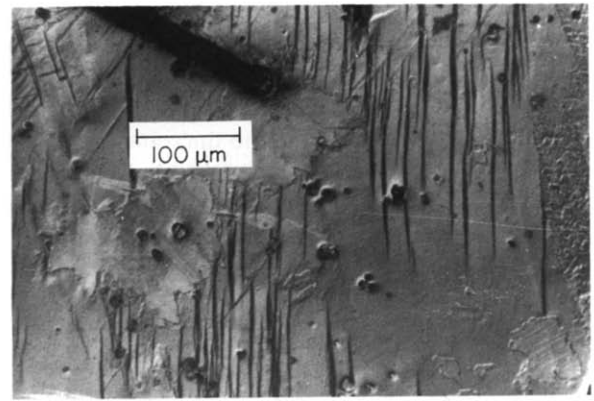
(b)

Fig. 4. (a) Low and (b) high resolution optical micrographs of water-quenched U-0.43at.%W after 1 h at room temperature: appearance of the first lenticular  $\alpha$ -U platelets within the  $\beta$ -U matrix.

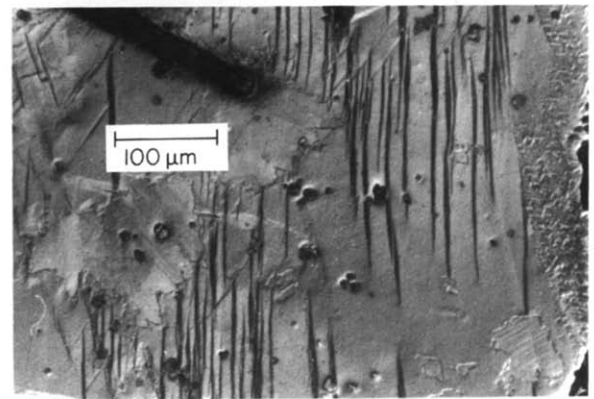
martensitic transformation has been observed in several uranium-rich alloy systems. Various aspects of this transformation have been studied in uranium-transition metal systems, in particular U-Cr [1] and U-Ga [3,15]. The results of the present study indicate that at room temperature tungsten clearly appears to retain a metastable  $\beta$ -U structure. This structure then transforms at room temperature by means of a complex transformation which has some of the characteristics of a martensitic transformation but also some definite isothermal components into a supersaturated  $\alpha$ -U solid solution. A detailed study of the uranium-rich side of the U-W system, especially the shear-like transformation, its isothermal features and its dependence upon the tungsten content, is currently in progress and will be reported later.

#### 4. Conclusions

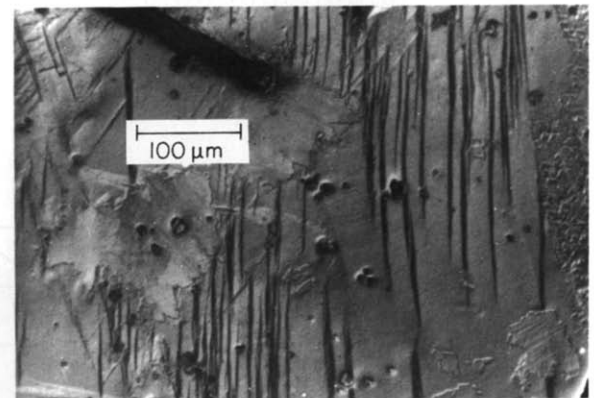
The solubility of tungsten in  $\gamma$ -U at 1060 °C was found to be about 1.0 at.%. Uranium alloys containing



(a)



(b)



(c)

Fig. 5. Sequence of micrographs illustrating the various stages of development of martensitic platelets in U-0.43at.%W alloy as a function of the time elapsed since the sample had been quenched from the  $\gamma$  phase range to room temperature: (a) after 3.5 h; (b) after 4 h; (c) after 5 h. Electropolished and etched using electrolyte containing 50 g chromic acid, 420 cm<sup>3</sup> acetic acid and 60 cm<sup>3</sup> water. In order to create the appropriate condition for polishing and etching, 20 V for 20 s and 3 V for 5 s respectively were applied.

about 1.0 at.% W allow the retention of the  $\gamma$ -U structure at room temperature. Smaller additions of the order of 0.43 at.% W allow the retention of the  $\beta$ -U structure at room temperature as a metastable structure. The metastable  $\beta$ -U alloy transforms at room temperature by a shear-like transformation with isothermal char-

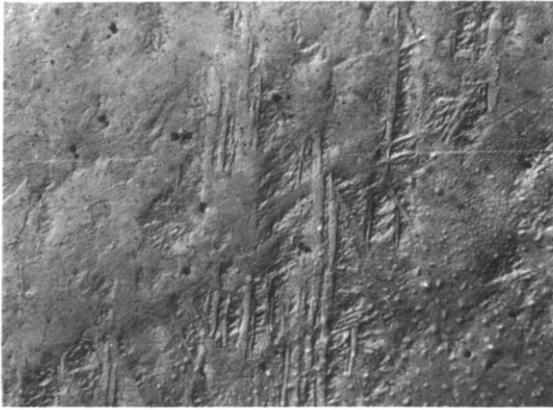


Fig. 6. Strong surface relief associated with the formation of martensitic platelets.

acteristics into a strained but undistorted variant of the  $\alpha$ -U structure.

#### Acknowledgments

We gratefully acknowledge the able technical assistance of S. Nathan, E. Nehama and Z. Barkai.

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