The unit cell of U₂Ru.

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Introduction

Of the six compounds reported in the uranium–ruthenium system (Dwight, 1961; Rough & Bauer, 1958) the structure of only URu₃ is known (Heal & Williams, 1955). In order to investigate this system further a preliminary study of U₂Ru was undertaken.

Experimental procedure and results

A melt containing 17·5 wt.% Ru (calc. for U₂Ru, 17·52%) was prepared, powdered, and then annealed at 870 °C., for 40 hr. Debye–Scherrer photographs were taken with Cu $K\alpha$ radiation. Attempts at indexing these patterns were unsuccessful, although metallographic examination indicated the original melt to be a single phase. Single crystal methods were suggested.

The Laue symmetry was found to be C_{2h}. Equi-

Table 1. *X-ray powder data for* U₂Ru

Q_o	hlk	Q_c	$\varDelta \times 10^4$
0·9048	331	0·9040	8
0·9222	{ 12,0,$\bar{2}$	0·9203	
	{ 12,0,1	0·9227	
0·9541	024	0·9549	−8
0·9888	{ 913	0·9879	
	{ 33$\bar{2}$	0·9875	
1·0290	31$\bar{5}$	1·0282	8
1·0906	62$\bar{4}$	1·0906	0
1·1232	315	1·1238	−6
1·1650	33$\bar{3}$	1·1650	0
1·2046	{ 12,2,0	1·2051	
	{ 12,2,$\bar{1}$	1·2043	
1·2225	333	1·2224	1
1·2434	624	1·2436	−2
1·2792	{ 12,2,1	1·2808	
	{ 12,2,$\bar{2}$	1·2783	
1·2930	12,0,$\bar{4}$	1·2927	3
1·3472	931	1·3476	−4
1·3568	91$\bar{5}$	1·3571	−3
1·4038	15,1,$\bar{1}$	1·4038	0
1·433†	{ 040	1·4307	
	{ 12,2,2	1·4314	
	{ 606	1·4429	
1·4681	041	1·4682	−1
1·5000	15,1,1	1·4994	6
1·5320	93$\bar{3}$	1·5323	−3
1·5807	042	1·5806	1
1·5990	{ 12,0,4	1·5989	
	{ 625	1·5997	
1·6073	15,1,$\bar{3}$	1·6077	−4
1·6430	{ 640	1·6432	
	{ 915	1·6442	
1·659†	(12,2,$\bar{4}$	1·6511	
	{ 12,2,3	1·6569	
	{ 15,1,2	1·6597	
	(64$\bar{1}$	1·6615	

† Broad, poorly resolved lines

$$Q_c = h^2 a^{*2} + k^2 b^{*2} + l^2 c^{*2} + 2hla^*c^* \cos \beta^* + D \sin^2 2\theta$$
$$D = (-10·2 \pm 3·2) \times 10^{-4}$$

Wave lengths used:

Cu $K\alpha_1 = 1·54050$, Cu $K\alpha_2 = 1·54434$ Å .

inclination Weissenberg photographs were taken, using Cu $K\alpha$ radiation, about the a axis and the b axis. With the aid of approximate lattice parameters calculated from rotation and Weissenberg photographs, and the observed intensities, the strongest lines of the powder pattern were indexed. A sin² 2θ absorption correction term was included and the parameters refined and standard deviations estimated by least-squares. Reflections with $Q (= 4 \sin^2 \theta/\lambda^2) < 0·9 (\theta < 47°)$ were not used. A comparison of observed and calculated Q values is given in Table 1. The final lattice parameters, with their standard deviations are:

$$a = 13·106 \pm 0·0014, \quad b = 3·343 \pm 0·0002,$$
$$c = 5·202 \pm 0·0009 \text{ Å}; \quad \beta = 96° 9·6' \pm 2·8'.$$

The calculated density for $Z = 4$ is 16·92 g.cm.⁻³ (observed, 17·00 g.cm.⁻³).

In view of the high absorption of U₂Ru and the irregular shape of the crystal used for the Weissenberg photographs (the best crystal found was 0·5 × 0·2 × 0·1 mm. in size) it is probable that the observed intensities do not possess a high degree of accuracy. In consideration of this uncertainty in the intensities it was decided to go
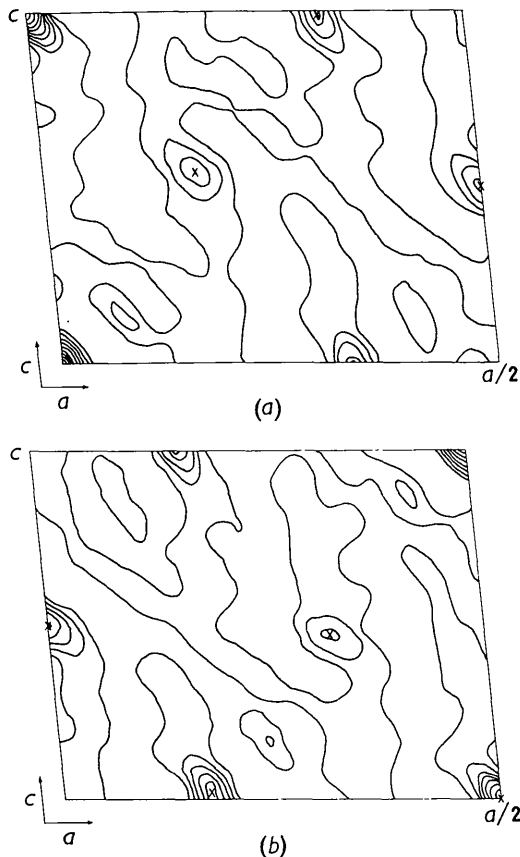

Fig. 1. (a) *H*0*L* Harker section. (b) *H*½*L* Harker section.

through only the preliminary stages of a structure determination in order to arrive at an approximate structure, and not to perform a complete structure determination.

If this structure contains mirror planes then packing arguments dictate that all atoms must lie in these mirror planes. (The metallic radii, for coordination number 8 (Pauling, 1947), are: U, 1·463 Å, Ru, 1·283 Å). This is consistent with the observation, from the Weissenberg photographs, that the $h0l$ layer is identical in appearance to the $h2l$ layer but not to the $h1l$ layer. If the space group is assumed to be centrosymmetric then it must be either $P2/m$, with both mirror planes having the same atomic composition (since $F(0k0)$ was observed $= 0$, for k odd), or $P2_1/m$.

$H0L$ and $H\frac{1}{2}L$ Harker sections were calculated using an IBM 650 computer and are shown in Fig. 1(a) and (b). (In both Figs. contours are drawn at the same equal arbitrary intervals. Negative contours are omitted.) A possible solution can be obtained by placing (unidentified) atoms at roughly

$$(x, y, z;\ x, y+\tfrac{1}{2}, z+\tfrac{1}{2}) + ((n/3), 0, 0;\ (2n+1)/(6), 0, \tfrac{1}{2}),$$
$$n = 0, 1, 2.$$

This arrangement of atoms may be placed into $P2/m$ by putting a 2-fold axis at $x = 0$, $z = 0$ or $x = \tfrac{1}{12}$, $z = \tfrac{1}{4}$, or into $P2_1/m$ by putting a 2-fold screw axis at $x = \tfrac{1}{12}$, $z = 0$ or $x = \tfrac{1}{6}$, $z = \tfrac{1}{4}$. These four cases are exhaustive. In order to locate the atoms of U and Ru structure factors (trigonometric part only) were calculated for all possible ordered arrangements, consistent with the symmetry,
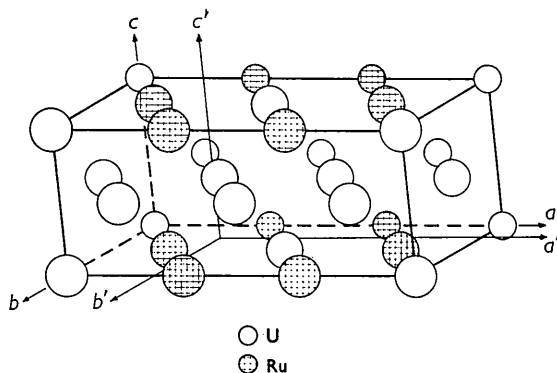

Fig. 2. Suggested atomic packing in U$_2$Ru. (The size of the circles represents relative distance from the observer.)

for each case, and compared qualitatively with the observed data.

The atomic assignments shown in Fig. 2 give the best agreement between the calculated and observed structure factors (Table 2). This configuration can be placed into $P2/m$ by placing the origin at $x = y = z = 0$ (axes abc) or into $P2_1/m$ by placing the origin at $x = \tfrac{1}{12}$, $y = -\tfrac{1}{4}$, $z = 0$ (axes $a'b'c'$). However, the above structure is only approximate as suggested by the Harker sections and as evidenced by the fact that the given atomic positions lead to a calculated intensity of zero for reflections with

Table 2. *Qualitative comparison of observed and calculated structure factors*

Class of reflections		$|F_o|$	$|F_c|$
$0k0$ k_{odd}		0	0
k_{even}		vs	912
hkl $(h+k)_{odd}$		vw	0
k_{even}	$h = 6n$, l_{even}	vs	912
	$h = 6n$, l_{odd}	m	192
	$h = 6n \pm 2$	w	96
k_{odd}	$h = 6n+3$, l_{even}	m	192
	$h = 6n+3$, l_{odd}	vs	912
	$h = 6n \pm 1$	w	96

$h + k = 2n + 1$ whereas a few are observed as very weak reflections.

The structure may be considered to consist of approximate layers of atoms parallel to (001). Alternate layers contain only U atoms with the intermediate layers having the atomic composition $\tfrac{1}{3}$U, $\tfrac{2}{3}$Ru.

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The determination of crystallite size from diffraction profiles using standard deviation as a measure of breadth.*

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Methods of X-ray crystallography provide a suitable means of obtaining information about the internal structure of grains in photographic emulsions (Herz, 1960). In particular, the measurement of line broadening

can give information about the presence of small crystallites and lattice distortion (Waidelich, 1958). We report here the outcome of some preliminary investigations into this problem.

The unambiguous interpretation of measurements of line broadening is difficult, especially when both distortion and size effects are present together. This was shown