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The crystal structure of TiU_2.

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One intermediate phase exists at room temperature in the titanium–uranium system (Buzzard, Liss & Fickle, 1952) at a composition near 65 atomic % uranium. This phase was shown to correspond to a compound TiU_2, the structure of which has been determined from powder photographs.

The alloys were prepared by melting charges of 15–20 g. in an argon arc furnace. The uranium, which was provided by the Ministry of Supply, was of purity greater than 99·9%, and the titanium, an I.C.I. iodide material, contained about 0·3% total impurities. After arc melting, the specimens were heated in an evacuated silica tube for 4 weeks at 950° C. This annealing treatment was found to produce metallographically homogeneous samples with only a small degree of surface oxidation. The specimens were powdered by crushing and the resulting powders were annealed in small silica capsules at 600–650° C. for 24 hr. X-ray powder photographs were taken in a 9 cm. Hilger camera, using Cu $K\alpha$ radiation.

Table 1. *Diffraction line spacings and intensities for* TiU_2

hkl	d_c (Å)	d_o (Å)	I_c	I_o
100	4·067	4·067	32	*mw*
001	2·797	2·797	41	*mw*
110	2·375	2·381	200	*s*
101	2·321	2·319	109	*m*
200	2·056	2·056	21	*w*
111	1·823	1·825	211	*s*
201	1·670	1·667	84	*m*
210	1·568	1·570	36	*mw*
002	1·414	1·414	34	*w*
300	1·385	1·386	105	*ms*
211	1·374	1·375	118	*ms*
102	1·341	1·341	31	*w*
301	1·246	1·246	153	*s*
112	1·220	1·220	187	*s*
220	1·203	1·202	91	*ms*
202	1·171	1·171	25	*w*
310	1·156	1·155	26	*w*
221	1·108	1·108	129	*s*
311	1·071	1·072	108	*s*
212	1·054	1·054	51	*mw*
400	1·041	1·042	13	*vw*
302	0·9946	0·9933	192	*s*
401	0·9799	0·9792	58	*mw*
320	0·9573	0·9573	32	*w*
003	0·9472	0·9471	8	*vw*
103	0·9231	0·9236	70	*m*
222	0·9189	0·9189	256	*s*
410	0·9114	0·9114	259	*s*
321	0·9018	0·9077	152	*ms*
312	0·8980	0·8977	84	*m*
113	0·8819	0·8819	217	*s*
411	0·8680	0·8680	466	*vs*
203	0·8631	0·8632	105	*m*
402	0·8442	0·8414	58	*mw*
500	0·8360	0·8357	69	*mw*
213	0·8131	0·8132	324	*vs*
330	0·8045	0·8045	322	*vs*
501	0·8022	0·8021	189	*s*
322	0·7954	0·7952	260	*s*
420	0·7893	0·7893	145	*ms*
303	0·7841	0·7843	693	*vs*
331	0·7751	0·7751	1220	*vs*

vs=very strong, *s*=strong, *ms*=medium strong, *m*=medium, *mw*=medium weak, *w*=weak, *vw*=very weak.

All spacings are in true Ångström units.

Forty-two lines were measured in the powder pattern and indexed on the basis of a hexagonal unit cell. No systematic absences were observed. A faint extra pattern noted in some films was identified as UO_2. Accurate parameter values for the unit cell were obtained by using Nelson & Riley's function (Nelson & Riley, 1945) and plotting ($hk0$) and ($00l$) lines for the determination of a and c respectively.

In Table 1 are listed the indices assigned to the reflexions and the calculated and observed planar spacings. The calculated values of d were derived from the expression

$$\frac{1}{d^2}=\frac{4}{3}\cdot\frac{h^2+hk+k^2}{a^2}+\frac{l^2}{c^2}\,,$$

appropriate values of a and c being obtained from the Nelson & Riley plots.

The calculated and observed intensities for the postulated $C32$ structure type are also listed in Table 1. The intensity comparison substantiates the structure. The calculated intensities were computed by the formula

$$I \propto \frac{1+\cos^2 2\theta}{\sin^2\theta\cos\theta}\,p|F|^2A(\theta)\,,$$

where $(1+\cos^2 2\theta)/\sin^2\theta\cos\theta$ is the combined Lorentz and polarization factor, p is the planar multiplicity factor, F is the structure factor, and $A(\theta)$ is the absorption factor.

The structure proposed on the basis of these results is accordingly as follows:

Space group: D^1_{6h}–$P6/mmm$; 3 atoms per unit cell;
a = 4·828 Å, c = 2·847 Å;
1Ti in (a): 0, 0, 0;
2U in (d): $\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$; $\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$;
structure type $C32(AlB_2)$.

TiU_2 is a layer-type structure with alternate planes of titanium and uranium atoms normal to the c axis. Each titanium atom is surrounded by twelve uranium atoms, six in the layer above and six in the layer below, at a distance of 3·14 Å, and two titanium atoms at 2·85 Å. Each uranium atom is surrounded in the uranium plane by an equilateral triangle of three uranium atoms at 2·80 Å, and by two uranium atoms, one in the plane above and one below at 2·85 Å.

Other phases crystallizing in the AlB_2 structure are: the diborides of Al, Cr, Nb, Ta, Ti, V and Zr (Kiessling, 1950); $CaGa_2$, $LaGa_2$ and $CeGa_2$ (Laves, 1943); UHg_2 (Rundle & Wilson, 1949); β-USi_2 (Zachariasen, 1949); $BiIn_2$ (Makarov, 1949); $PtZn_2$ and $PdZn_2$ (Nowotny, Bauer, Stempfel & Bittner, 1952); and $PdHg_2$ (Nowotny, Stempfel & Bittner, 1952).

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The preparation of a $K\beta$ filter for X-ray crystallography with special reference to chromium radiation.

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The ideal $K\beta$ filter consists of the pure element rolled to a thin foil (Ross, 1928). Where the element is not ductile (or not available) alternative methods have been adopted: (1) A compound of the filtering element with others of low atomic number is spread on a piece of paper coated with shellac, or suspended in a paraffin block, which is afterwards cut to the calculated thickness (Pierce, 1931). (2) Filter paper is soaked in a solution of a compound of the filtering element, and dried. (3) The element is electro-deposited on aluminium foil (Wood, 1931). (4) The compound is mixed with beeswax and pressed between two rocksalt cleavage surfaces at 50° C. The sodium chloride is then removed by dissolution in water (Kratky, 1943). (5) The compound is kneaded uniformly into high-melting-point paraffin wax, and pressed between spaced glass plates (Kirkpatrick, 1942). (6) The filter substance is suspended in collodion, spread out in a thin sheet, and allowed to dry (Sidhu, 1937*a*, *b*).

This last method has been developed here in order to obviate the use of expensive commercial vanadium pentoxide filters. The fact that we have reduced the amount of supporting material used without losing strength and flexibility of the filters, gives them particular value where fourth-period elements (e.g. Ti, V, Cr, Mn and perhaps Co) are involved.

Vanadium tetroxide was prepared by heating pure ammonium metavanadate (analysed for heavy metals) in a covered platinum crucible, using a Bunsen burner. A blue-black product was obtained consisting of vanadium tetroxide (80–90%) and vanadium trioxide (10–20%), the actual composition depending on the time of heating. Prolonged heating favoured the formation of the tetroxide or even the pentoxide. The pentoxide-free product, obtained by strong heating for half an hour, was used here. The following recipe is based on several experiments: For a filter of 24 cm.^{2} area, 0·37 g. 'V_2O_4' was employed, i.e. 5% more than the amount corresponding to 0·009 g. vanadium per cm.^{2}. Prolonged dry grinding in an agate mortar followed by grinding after addition of ethyl acetate eliminated granular matter. 24 drops (i.e. 0·45 g. containing 0·13 g. dry matter) of ethyl acetate–pyroxylin lacquer and half a drop of dibutyl phthalate were added, the latter to make the film flexible. The consistency was made that of a thin gruel by further addition of ethyl acetate, and the mixture was then poured on to a glass plate of area 24 cm.^{2}, spread evenly with a paint brush, and dried in the air. After one night it was possible to strip off the filter with a wet razor blade. It was strengthened by pasting on to it a sheet of condenser paper (weighing 1 mg.cm.$^{-2}$). Application of larger amounts of dibutyl phthalate was unnecessary and impeded the drying. An analysis of the filters indicated that they tended to contain too much pyroxylin near the edge of the glass plate; therefore only the central portion of the plate was used.

In order to compare the different $K\beta$ filters for chromium radiation, the increase in exposure time and the factor reducing the intensity ratio, I_β/I_α, relative to no filters, have been calculated (Table 1). Four different filters have been considered: the metal foil, our 'V_2O_4' filter, an ammonium metavanadate filter (as recommended by Sidhu (1937*a*, *b*)), and the commercial filter. The metavanadate filter contains the same percentage of pyroxylin as ours (i.e. $< 40\%$) while the commercial filter contains $> 80\%$ pyroxylin. Furthermore, the commercial product had only 5·5 mg. vanadium per cm.^{2}.

Table 1. *Comparison of filters*

	Prolongation factor	Reduction factor on I_β/I_α
No filter	1	1
Vanadium foil	2·0	1/53
'V_2O_4' filter	3·0	1/47
NH_4VO_3 filter	4·2	1/44
Commercial filter	6·6	1/11

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