

of a glass rod, subsequent contraction of the adhesive upon cooling will result in misalignment of the crystal. If the crystal is mounted on the end of a platinum wire, by heating the end of the wire and inserting it into the crystal, the alignment difficulty is overcome, but a temperature gradient will be present in the crystal when it is cooled, since the wire will conduct heat from the goniometer head to the crystal.

The slit of the layer-line screen is covered by a thin strip of cellophane; the collimator is inserted through a small hole in the cellophane.

The film holder is made of two halves and can thus be put on and taken off without disturbing the temperature distribution in the apparatus.

In the case of research on a substance melting below room temperature, the liquid is also put into a thin-walled glass capillary and slowly frozen by the stream of cold gas. If necessary, a single local counter-current of dry air at room temperature can be introduced through a glass tube with a narrow nozzle inserted through the cellophane of the layer-line slit.

By adjusting the rates of flow of both gas streams it is possible to control the rate of crystallization very sharply. If the beam catcher is turned out of the way, the crystallization process may be viewed through a small microscope, mounted on the Weissenberg chassis. This is readily converted into a polarizing microscope by means of a pair of polarizers mounted on a yoke which bridges the layer-line screen.

There is a slot in the top of this screen extending along a part of its length, and ordinarily closed by a

sliding cover. If this slot is opened the beam catcher may be turned and adjustments of the goniometer head may be made as well.

For making diagrams above room temperatures, a long and narrow heating coil is introduced into the Dewar tube, insulated from the leads of the thermocouple. This coil heats the stream of nitrogen gas and thus the crystal.

The capillary containing the crystal is inserted into a closely fitting hole drilled in the end of a copper rod, which in turn is mounted on the goniometer head.

Until now only rotation and oscillation photographs have been made at temperatures between room temperature and 300° C.

Weissenberg photographs have not been made at these higher temperatures since the layer-line screen would be heated by the hot gas. If the layer-line screen is not present, however, the hot gas passes out through the end of the camera without warming the film excessively.

A Weissenberg camera similar to that described above is now commercially offered by 'Nonius', Delft, The Netherlands.

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The crystal structure of WAl_5 . By J. ADAM and J. B. RICH, *Atomic Energy Research Establishment, Harwell, Didcot, Berks., England*

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The crystal structure of WAl_{12} was recently investigated by Adam & Rich (1954). In the present communication we describe the crystal structure of the δ W-Al intermetallic compound. According to the phase diagram of Clark (1940) this phase should be of approximate composition W_2Al_9 ; the present structure determination shows, however, that the ideal formula unit is WAl_5 .

The unit cell and systematic absences of the X-ray reflexions were determined from rotation, oscillation and Weissenberg photographs. The measurements of accurate unit-cell dimensions and intensities were made on powder photographs taken in a precision 19 cm. camera (Adam, 1954) using filtered $Cu K\alpha$, and $W L\alpha$ radiations. (The $W \beta$ radiation can be effectively removed by a Cu foil 0.001 in. thick.)

The use of $W L\alpha$ radiation is of great advantage in the Bragg-angle range $20^\circ < \theta < 45^\circ$. The resolution of the $Cu K\alpha$ doublet is imperfect in this range, thus reducing greatly the accuracy of measurement of interplanar spacings. On the other hand the $W L\alpha$ doublet is well resolved at fairly low angles and in addition the intensity of the α_2 component is much less than that of the α_1 . The range of θ below 45° is important in lattice-parameter measurements of non-cubic substances when the

unit cell is fairly large and comparatively few of the high-angle lines are free from overlap.

Intensities of single-crystal reflexions were estimated visually. The assessment of the absorption correction presented considerable difficulty. The method of Howells (1950) was tried, and, although good qualitative agreement was obtained between observed and calculated intensities for reflexions in each zone, the overall agreement was not quite satisfactory. Powder intensities were therefore used as a final check of the structure. Integrated intensity measurements were made on 45 powder lines, using a Hilger recording microphotometer. The absorption factor taking into account the particle absorption (Taylor, 1945; Brindley, 1945; de Wolff, 1951) was used, and the temperature correction and scale factors were obtained from a plot of $\log(I_c I_0)$ against $(\sin \theta/\lambda)^2$.

All observed X-ray reflexions were indexed on the basis of an hexagonal unit cell with

$$a = 4.9020 \pm 0.0003, \quad c = 8.8570 \pm 0.0005 \text{ \AA}$$

($\lambda Cu K\alpha = 1.54050$ and $\lambda W L\alpha = 1.47634 \text{ \AA}$).

The only observed space-group extinctions were 0001 with 1 odd. The three possible space groups are therefore C_6^h , C_{6h}^2 and D_6^h , and the number of atoms per unit cell

Table 1. *Calculated and observed powder intensities for* WAl_5

<i>hkl</i>	I_c	I_o	<i>hkl</i>	I_c	I_o
0002	161	176	30 $\bar{3}$ 2	102	97
10 $\bar{1}$ 0	113	116	21 $\bar{3}$ 4	47	44
10 $\bar{1}$ 1	574	624	11 $\bar{2}$ 6	266	275
10 $\bar{1}$ 2	135	143	22 $\bar{4}$ 0	64	59
11 $\bar{2}$ 0	287	288	22 $\bar{4}$ 1	} 96	} 101
10 $\bar{1}$ 3	264	243	10 $\bar{1}$ 7		
11 $\bar{2}$ 1	69	78	20 $\bar{2}$ 6		
0004	211	245	30 $\bar{3}$ 4	} 417	} 417
11 $\bar{2}$ 2	} 921	} 925	21 $\bar{3}$ 5		
20 $\bar{2}$ 0			22 $\bar{4}$ 2		
20 $\bar{2}$ 1	196	185	31 $\bar{4}$ 1	112	101
10 $\bar{1}$ 4	58	51	0008	44	50
20 $\bar{2}$ 2	56	48			
11 $\bar{2}$ 3	43	42	31 $\bar{4}$ 3	92	117
20 $\bar{2}$ 3	138	127	20 $\bar{2}$ 7	} 95	} 109
11 $\bar{2}$ 4	} 375	} 360	21 $\bar{3}$ 6		
10 $\bar{1}$ 5			22 $\bar{4}$ 4		
			10 $\bar{1}$ 8	} 64	} 73
21 $\bar{3}$ 0	41	34	30 $\bar{3}$ 6		
21 $\bar{3}$ 1	222	201	11 $\bar{2}$ 8	} 107	} 131
20 $\bar{2}$ 4	35	37	22 $\bar{4}$ 5		
21 $\bar{3}$ 2	67	66	21 $\bar{3}$ 7	94	104
0006	21	21	20 $\bar{2}$ 8	} 110	} 122
11 $\bar{2}$ 5	23	25	31 $\bar{4}$ 5		
30 $\bar{3}$ 0	} 408	} 401	32 $\bar{5}$ 1	94	106
21 $\bar{3}$ 3			22 $\bar{4}$ 6	190	212
10 $\bar{1}$ 6	28	25	41 $\bar{5}$ 2	375	390
20 $\bar{2}$ 5	78	76	30 $\bar{3}$ 8	} 280	} 303
			1,0, $\bar{1}$,10		
			31 $\bar{4}$ 7	} 136	} 149
			40 $\bar{4}$ 6		

must be even. Assuming that the volumes occupied by individual atoms in an alloy are not greatly different from those occupied by these atoms in elements, the number of atoms per cell in the δ phase is approximately 12. This suggests that the ideal composition of the δ phase is WAl_5 with two formula units per cell, rather than W_2Al_6 , as would appear from the phase diagram.

The calculated density of the sample is 5.71 g.cm.^{-3} . The value obtained experimentally on a small quantity of powder was $\rho = 5.5 \text{ g.cm.}^{-3}$.

Spatial considerations rule out space group D_6^h . The choice of space group is therefore reduced to the centrosymmetric C_{6h}^2 and non-centrosymmetric C_6^2 .

Packing considerations lead to two simple possible atomic arrangements in close-packed layers, one centrosymmetric and another non-centrosymmetric. Intensities of X-ray reflexions were calculated for both. Comparison with observed intensities showed good agreement for the non-centrosymmetric space group $C_6^2-P6_3$, and large discrepancies for the centrosymmetric structure.

Assuming a close-packed layer structure, atomic positions are:

$$\begin{aligned}
 &2 \text{ W } 2(b): \frac{1}{3}, \frac{2}{3}, z; \frac{2}{3}, \frac{1}{3}, \frac{1}{2}+z; \text{ with } z = \frac{1}{2}. \\
 &2 \text{ Al } 2(b): \frac{1}{3}, \frac{2}{3}, z; \frac{2}{3}, \frac{1}{3}, \frac{1}{2}+z; \text{ with } z = 0. \\
 &2 \text{ Al } 2(a): 0, 0, z; 0, 0, \frac{1}{2}+z; \text{ with } z = 0. \\
 &6 \text{ Al } 6(c): x, y, z; \bar{y}, x-y, z; y-x, \bar{x}, z; \bar{x}, \bar{y}, \frac{1}{2}+z; \\
 &\quad y, y-x, \frac{1}{2}+z; x-y, x, \frac{1}{2}+z; \\
 &\quad \text{with } x = y = \frac{1}{3}, z = \frac{1}{4}.
 \end{aligned}$$

Comparison between calculated and observed intensities is shown in Table 1. The agreement residual is $R = 0.06$.

The structure of WAl_5 is a close-packed layer structure with four types of layer stacked in the order



Layers A and A' contain both aluminium and tungsten atoms, layers B and C consist entirely of aluminium atoms. The interatomic distances are given in Table 2.

An alloy of composition $MoAl_5$ is reported in the literature (see Wachtell, 1952). An X-ray powder photograph obtained by us from this alloy was very similar to that of WAl_5 : there were only slight differences in relative intensities of some of the lines. The two crystal structures, therefore, are probably identical or at least very similar.

Table 2. *Interatomic distances in* WAl_5

Atom	Number and kind of neighbours	Interatomic distance (Å)
Al in layer A or A'	3 Al in same layer	2.83
	3 W in same layer	2.83
	3 Al in layer B	2.75
	3 Al in layer C	2.75
Al in layer B or C	6 Al in same layer	2.83
	2 Al in layer A	2.75
	2 Al in layer A'	2.75
	1 W in layer A	2.75
	1 W in layer A'	2.75
W in layer A or A'	6 Al in same layer	2.83
	3 Al in layer B	2.75
	3 Al in layer C	2.75

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