Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

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A Weissenberg camera for use at constant temperatures between about −150° C. and 300° C.

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The apparatus has been developed more especially for use at low temperatures. A stream of cooled gas is blown against the crystal through a long internally silvered Dewar tube (Fig. 1(*a*)). The inner wall of this tube is not of uniform width but has a number of wider sections, *a*, in order to ease the tension caused by thermal contraction with respect to the outer tube. The end of the tube is pushed quite close to the crystal and the temperature at this end is measured by means of a copper–constantan thermocouple, constructed of very thin wires which are led in through the Dewar tube. This permits continuous control of the temperature.

After cooling the crystal, the cold gas leaves the camera by passing between the outer wall of the Dewar tube and the layer-line screen. Ice formation is avoided by heating the gas with a small heating coil, *b*, (Fig. 1(*b*)), consisting of five turns of 0·5 mm. Nichrome wire, which is mounted on the end of the Dewar-tube carrier, which in turn is inserted in a long layer-line screen, *c*. This screen extends over the whole length of the camera and is held in place by a special support, *d*, on the end of the Weissenberg chassis.

The Dewar tube is centred in the layer-line screen by two perforated pertinax rings, held in position by metal rods, *e*, through which the current for the heating coil also flows.

The cooling gas can consist of evaporated liquid air, the flow of which can be regulated by a heating spiral in it. A more economical method is to use gaseous nitrogen, which is pre-cooled and at the same time dried by being passed through traps immersed in a mixture of dry ice and alcohol. The nitrogen is then passed through a helix, constructed of 5 mm. internal diameter copper tubing, which is kept immersed in liquid air.

If cooling by liquid air is omitted, this method is satisfactory also for obtaining temperatures down to −70° C. The pressure of the incoming gas was measured accurately by means of a liquid manometer, and adjustments were made as needed to maintain a constant pressure.

It is essential to mount the crystal in a thin-walled capillary in order to cut down the heat flow from the goniometer head to the crystal.

If an adhesive is used to fasten the crystal to the end

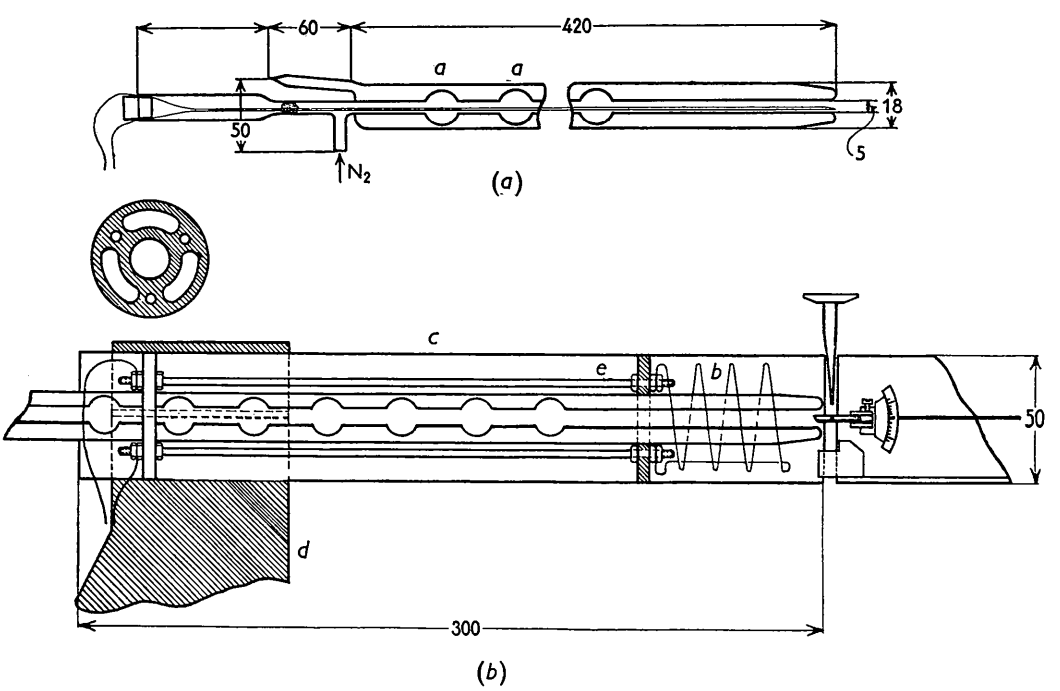

Fig. 1. (*a*) High-vacuum Dewar tube, silvered on the inside, showing the expansion rings. (*b*) Layer-line screen with Dewar tube in carrier and heating coil. Dimensions are in millimetres.

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.

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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 β 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 $20° < \theta < 45°$. 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 $\log (I_c I_o)$ against $(\sin\theta/\lambda)^2$.

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

$$a = 4{\cdot}9020 \pm 0{\cdot}0003,\ c = 8{\cdot}8570 \pm 0{\cdot}0005 \text{ Å}$$

(λ Cu $K\alpha$ = 1·54050 and λ W $L\alpha$ = 1·47634 Å).

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