

METALLURGY

High-Temperature Phase Transformation in Beryllia

A HIGH-TEMPERATURE X-ray diffractometer has been used to investigate a suggestion by S. B. Austerman (private communication) that there was a reversible phase transition in beryllia at $2,050^{\circ}\text{C}$. By this means evidence has been obtained of a new phase appearing at $2,080 \pm 50^{\circ}\text{C}$. on heating, which reverted to the usual hexagonal beryllia structure at $1,980 \pm 50^{\circ}\text{C}$. on cooling. In each case the phase-change took place over some 30°C .

The interplanar spacings of the new phase recorded over the angular range of 35° – 140° 2θ , using copper $K\alpha$ radiation, are given in Table 1.

Table 1. INTERPLANAR SPACINGS OF HIGH-TEMPERATURE BeO PHASE

d (Å.)	Intensity	d (Å.)	Intensity
2.370	10	1.152	<1
2.120	8	1.118	<1
1.676	4	1.062	<1
1.500	1	0.898	<1
1.370	6	0.882	<1
1.316	<1	0.868	<1
1.187	3	0.840	<1

This table includes all the reflexions obtained at the high temperature, with the exception of those attributed to a deposit from the tungsten element of the furnace, which were present at all temperatures and increased in intensity with time. The only reflexion common to both the phases is the (101) reflexion of the original beryllia structure, which becomes the second recorded line of the new phase.

The high-temperature phase seems to have pronounced cubic characteristics; further experimental results are being obtained in the higher d -value range to enable a full structural analysis to be made.

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CRYSTALLOGRAPHY

Fourier Projections of Twinned Crystals

TWINNING of crystals often results in the superposition of reciprocal lattice points and the appearance of a pseudo-lattice. In many cases this can be recognized from the diffraction data by the presence of systematic absences inconsistent with those absences expected from space-group symmetry. Because of the superposition of much of the diffraction data, exact intensities for each reciprocal lattice point cannot be obtained. If it is assumed that the twinning is on a gross enough scale, then the intensities of the superimposed reflexions will be proportional to the sum of the F^2 of the individual reciprocal lattice points comprising the reflexion, without regard to their relative phases. Thus it is not possible to apply the accepted Fourier methods of crystal structure determination in the usual way. In the case of contact twins a physical separation can be made, and single-crystal data can be obtained; but in the event of fine polysynthetic twinning or the 'anti-phase domain' type of twinning usually resulting from cooling through a phase change, one is forced to work with the twinned material.

If it is assumed in a given sample that each portion of the twin is present in similar amounts, then a 'Patterson projection', either on the plane perpendicular to the twin axis or on one of the planes perpendicular to the twin plane, can be constructed using the pseudo-cell and the observed composite intensities. The 'Patterson map' so produced is identical to that obtained by superimposing, in the correct twin orientation and with their origins coincident, two 'Patterson maps' of the single crystal. It is then possible to transform this map from vector space to real space by the Buerger 'minimum method'.¹ If the image-seeking vector chosen is one joining two atoms related by a centre of symmetry, then the 'minimum map' produced will be equivalent to two superimposed electron-density maps of the crystal with their symmetry centres coincident.

These relations have been successfully tested on the known structure of orthorhombic CrB , and are at present being verified on monoclinic FeAsS .

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¹ Buerger, M. J., *Acta Cryst.*, 4, 531 (1951).

X-Ray Analysis of Parallel Growth and Disorder in Silicon Carbide Crystals

It is well known that silicon carbide forms a great variety of polytypes. The significance of these polytypes is so far not fully understood, although the known types appear to be consistent with the theory postulated by Ramsdell and Kohn¹ according to which: "Each polymer is characterised by a particular temperature stability range. At a given characteristic temperature a single type of polymer would produce a 'pure type' If stability ranges overlap two polymers might exist simultaneously resulting in a 'mixed type'".

In the course of our analysis of a number of silicon carbide single crystals by X-rays, some interesting observations have been made in the case of two crystals which might throw more light on the growth mechanism in silicon carbide and perhaps provide some further evidence on the formation of polytypes. The crystals, kindly lent to us by Prof. S. Tolansky, had been obtained commercially, and no details of their production were known.

One of the two crystals was of a light green colour and had grown with the (0001) face plane, roughly hexagonal in outline and about 10 mm. across. The crystal was wedge-shaped and tapered from 3 m. to a knife edge along one side of the hexagon. It had presumably grown by deposition on to a cooled flat surface. The plane (0001) face would then be next to the cooled surface. The top face was rounded. The second crystal was a thin (0.25 mm.), colourless, parallel-sided plate about 2 mm. square, the two sides of the plate being the (0001) pinacoid faces. There was no indication of the method of growth.

The glancing angle, α -axis oscillation photographs from the flat face of the large light-green crystal showed horizontal streaks on the non-zero layers. These corresponded to weighted rods in reciprocal space passing through the lattice points, parallel to c^* . Fig. 1 shows the horizontal streak on the first layer lines produced by such rods, which arise from random stacking of structural layers in the c direction.