Program details and uses

The main body of each program is written in Elliot 803 autocode while the graph plotter routines are in machine code. Because of the relatively small storage of the Elliot 803 computer, two programs were necessary to cover all the desired methods of output. With program A all the atoms are plotted as circles with either constant radius, or radii proportional to their height above the plane of projection. Program B considers slices through A and only atoms intersected by a plane parallel to, and at a specified height above, the plane of projection, are finally plotted. The atoms are again plotted as circles but their radii are dependent upon the atomic radii of the individual atoms and the intersection of the plane with the atoms. The data required for program A are as follows:

(1) \( a, b, c \) the lengths of the unit-cell edges;
(2) \( \alpha, \beta, \gamma \) the interaxial angles;
(3) \( h, k, l \) the indices of the plane of projection;
(4) \( N \) the number of atoms in the unit cell;
(5) \( \rho, \varphi, \lambda \) a number which determines whether to consider both +ve and -ve limits, or simply +ve limits;
(6) \( u, v, w \) the coordinates of the atoms in the unit cell;
(7) \( u_1, v_1, w_1 \) the coordinates of the atoms in the unit cell together with their radii.

The limits of the projection to be plotted in the X and Y directions.

With program B items 1-6 are the same. These are then followed by:

(8) \( X_1 \) to \( X_2 \), \( Y_1 \) to \( Y_2 \), \( 0 \) to \( Z_2 \) the limits of the projection to be plotted.

(9) \( N_3 \) the number of layers to be plotted.
(10) \( z_1 \), \( z_2 \) the heights of these layers above the plane of projection.

With the crystal structure is built up from more than one type of atom, if desired, the program can be repeated such that for each repeat the atom coordinates are those of a particular type of atom. If the colour of the ink is changed the location of each type of atom becomes evident. These programs have been found to be useful in the types of investigation already mentioned particularly when the unit cell contains a large number of atoms or when the plane of projection is not one with simple indices.

Fuller details of the programs are available on request from one of the authors (D.J. Dyson).

The authors wish to thank Dr F.H. Saniter, O.B.E., Director of Research, Midland Group, British Steel Corporation, for permission to publish this paper. They would also like to thank Mrs E.R. Denton and several of their colleagues for helpful discussions.

References


(Received 8 September 1969 and in revised form 15 January 1970)

A seven-step precision alignment procedure for the Kratky small-angle X-ray camera is described.

The small-angle X-ray camera based on the collimation geometry proposed by Kratky & Skala (1958) has found increasing use in the fields of metallurgy, physics, chemistry and biophysics. Effective use of this instrument requires that all slit edges in the collimation system be parallel to each other and to the line of sight of the focal spot and that the beam axis of the collimation system accurately extend through the focal spot.

A precision alignment procedure has therefore been developed for the camera. The procedure requires only a proportional counter to measure the X-ray intensity distribution in the incident beam as opposed to other methods which require fluorescent screens and X-ray film. The technique has been applied both to an unmodified Kratky camera and to one incorporating vacuum bellows seals and a diffracted-beam graphite monochromator (Hendricks, 1970). Further, it has been successfully used by I.S. Patel and P.W. Schmidt at the University of Missouri on the basis of its full written disclosure available in unpublished report form (Anderegg, Mardom & Hendricks, 1971). Dur-
The work, auxiliary slits were developed to aid in the procedure. These slits are a convenience rather than a necessity. They are especially useful for checking the camera alignment during routine operation, as their use does not require the partial disassembly of the camera as is the case with the alignment slits provided by the manufacturer.

The proffered alignment procedure consists of a sequence of seven operations, each of which must be undertaken in the proper order. These are:

1. Tilting the collimator parallel to the focal spot.
2. Accurately positioning the camera axis to pass through the center of the focal spot, as judged in both the horizontal and vertical directions.
3. Adjusting the jaws of the receiving slit to be parallel.
4. Tilting the receiving slit to be parallel to the collimated beam.
5. Tilting the specimen chamber to be parallel to the collimation system.
6. Setting the entrance and receiving slits to the appropriate dimension and recentering the collimator beam-axis on the focal spot.
7. Measuring the rocking curve and computing the weighting functions.

Various extra stages are required for the modified camera. Full details of the procedure for both cameras have been given elsewhere by the present authors (Anderegg et al., 1970).

References


(Received 28 November 1969)

X-ray powder data for the compounds CaO·Al₂O₃ and CaO·2Al₂O₃ have been obtained from the pure synthesized compounds. The detail of the patterns is considerably greater than that obtained by previous workers.

Introduction

The object of this work has been to prepare CA (=CaO·Al₂O₃) and CA₂ (=CaO·2Al₂O₃) and obtain their detailed X-ray powder diffraction patterns in order to resolve some anomalies which existed in the weaker lines of the previously published patterns.

The crystal structure of CA was determined by Dougill (1957) who published in a preliminary report a structure similar to β-tridymite—a three-dimensional arrangement of AlO₄ tetrahedra with Ca accommodated in the cavities. The space group was reported as P2₁/n. A review of the powder diffraction data has been made by Ampian (1964) who recommends for use the pattern obtained by the U.S. Bureau of Mines, which he publishes, and that of Yannacquis (1954). Neither of these patterns is indexed.

The crystallography of CA₂ has also been reviewed by Ampian, who recommends the work of Boyko & Wisnyi (1958) as being the most reliable. They show the space group to be C2/c and account for the apparent uniaxial optical behaviour by showing that there does exist a small difference between the α and β refractive indices. A powder pattern by Wisnyi is published in the ASTM Powder Data File (7-0082).

Experimental details

CA and CA₂ were prepared by heating stoichiometric mixtures of dried Analar calcite and a commercial Gibbsite (α-Al₂O₃ · 3H₂O) at 1400-1450°C for 5 hours in a vertical tube furnace to give about 5 g of the finished product. X-ray powder diffraction patterns were obtained using a Philips PW1310 2 kW X-ray generator with a PW1050 goniometer, a proportional counter and pulse-height discrimination. Filtered copper radiation (λ Cu = 1.54178 Å, λ Kα = 1.54051 Å) was employed. The CA preparation contained a small amount (~2 wt.%) of C₁₂A₇ and the CA₂ preparation a trace (~½ wt.%) of CA. A refined unit-cell was obtained from the powder data using the program of Marples & Shaw (1966) adapted for an Elliott 4130 computer. This program compares observed data with calculated data from the trial cell, indexes, and then refines the unit-cell lattice parameters using Cohen’s least-squares method with the Nelson–Riley extrapolation function. All lines have been indexed and satisfy the criterion that

|sin²θₐₐsb - sin²θₑₑₑₑ| < 0.0003.

Results

The unit-cells of CA and CA₂ are given in Table 1 and the powder diffraction patterns in Tables 2 and 3. The intensities are those observed on the diffractometer.

Table 1. Unit cells for CA and CA₂

<table>
<thead>
<tr>
<th></th>
<th>CA</th>
<th>CA₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Space group</td>
<td>P2₁/n (≡ P2₁/c)</td>
<td>C2/c</td>
</tr>
<tr>
<td>a</td>
<td>8.6975 ± 0.001 Å</td>
<td>12.888 ± 0.001 Å</td>
</tr>
<tr>
<td>b</td>
<td>8.092 ± 0.001</td>
<td>8.988 ± 0.001</td>
</tr>
<tr>
<td>c</td>
<td>15.208 ± 0.001</td>
<td>5.443 ± 0.001</td>
</tr>
<tr>
<td>β</td>
<td>90.14 ± 0.01</td>
<td>106.93 ± 0.01</td>
</tr>
<tr>
<td>Volume</td>
<td>1070.4 Å³</td>
<td>596.5 Å³</td>
</tr>
</tbody>
</table>
