Design and Fabrication of a 10 cm Diameter Back-Reflection Focusing Camera

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The design and fabrication of a high-temperature 10 cm back-reflexion symmetrically focusing camera is described. This camera was designed on the focusing principle. A tubular heater was attached to the sample for controlling its temperature to within \(\pm 2^\circ\)C. It is possible to measure precisely the lattice parameters of a powder sample with this camera and study their variation with temperature.

Introduction

Precise measurements of lattice parameters by means of X-rays give valuable information about thermal expansion coefficients of a material where conventional methods are inapplicable, such as when the physical state of the substance renders it unsuitable for bulk measurements, e.g. powders, porous solids.

A suitable camera for precise measurements of lattice parameter is a symmetrically focusing back-reflexion camera. This camera possesses nearly twice the resolving power of a Debye–Scherrer camera of the same radius and also requires a shorter exposure time than do other cameras. Therefore, a high-temperature symmetrically focusing back-reflexion camera of 10 cm diameter was designed and fabricated in this laboratory to measure accurate lattice parameters.

Modern commercial high-temperature cameras are available but they are often relatively expensive. Our camera is not only inexpensive but also efficient.

Design and fabrication

To bring the diffracted rays to a focus on the slit, the sample and the film should be on the circumference of the focusing circle. In the symmetrically focusing back-reflexion camera the slit and the sample are diametrically opposed. The geometry of the camera is illustrated in Fig. 1. A front elevation is given in Fig. 2(a) and a photograph in Fig. 3.

The camera has a tripod base (1) with levelling screws (2). A horizontal plate (3) is fixed to the tripod base through a round block (4). At one end of the horizontal plate a dove-tail block (5) is fixed with a vertical locating plate (6). The dove-tail block carries a screw arrangement for fine adjustment of the specimen. The specimen-carrying assembly (7) consists of a metallic ring (8) fixed vertically to the sliding block (9). The tube (10) holding the sample is fixed at the centre of the ring. This tube, which carries the sample assembly, can be given the desired vertical motion with the help of the screw arrangement (11) whenever necessary. The sample can be oscillated through a small angle about the camera axis with the help of a motor and cam arrangement. The other end of the horizontal plate (3) carries a vertical block. This block consists of a fixed piece (12) and a sliding piece (13). The sliding piece can be moved up and down by means of the screw (14). The base-plate (15) is fixed on the top of the sliding piece.

The film cassette (16) consists of an accurately machined cylindrical surface with gaps for incident and diffracted X-rays. The film is held tightly over the surface by means of a metal jacket. The film holder fits into a cylindrical enclosure which carries a collimator (17). The open end of the collimator has a diameter of 0.5 mm. This collimator hole limits the divergence of the beam incident on the powder specimen and stops the sample-holder assembly from being irradiated by the beam. By means of an optical method the axis of the collimator is made collinear with the axis of the specimen holder.

The film holder and the cylindrical enclosure are locked together by means of screws (18). The col-

Fig. 1. Geometry of the back-reflexion focusing camera.
Collimator is then inserted with its aperture towards the cylindrical surface. The face of the collimator, when pushed in, is completely in contact with the cylindrical surface which carries the film. The camera is then placed on the horizontal platform (15). So that it is not affected by the horizontal motion of the camera, it is placed in a groove on the horizontal platform (5). Its position on the horizontal platform is fixed by means of the guiding pins, which can be removed whenever necessary.

The specimen holder Fig. 2(b) consists of a quartz tube (1) over which a brass cap (2) is cemented. The brass cup (3), which carries the sample under study is fitted on this cap. A chromel–alumel thermocouple (4) is brazed on so that the tip of the thermocouple touches the inner surface of the cap (3) when placed in position.

For raising the temperature of the sample a tubular heater is used. The heating element consists of super Kanthal A wire which is covered with alumina which acts as an electrical insulator. The heater is fed with a stabilized AC supply and the temperature can be controlled to within $\pm 2^\circ$C by mean of a voltage stabilizer and a Variac. With this heater the temperature of the sample can be raised from room temperature to 500$^\circ$C. The temperature distribution along the axis of the heater was measured and it was found to be uniform except at the two open ends. A sample is heated for at least one hour before an exposure is made in order that it can attain the equilibrium temperature; this is checked by means of a sensitive galvanometer.

**Experimental**

The camera was calibrated by using standard substances (Rieck & Lonsdale, 1968) such as Al, W, NaCl and Pb(NO$_3$)$_2$ in powdered forms. The powder photographs of these samples were taken at different temperatures with Cu K$_\alpha$ radiation. The X-ray photographs were analysed by the standard procedure (Klug & Alexander, 1954; Edmunds, Lipson & Steeple, 1960). The precision of the final lattice parameters was found by the method of Jette & Foote (Jette & Foote, 1935). To verify these results the lattice parameters of these substances were also measured with a 11.46 cm Debye–Scherrer camera.

The lattice parameters of Al, W, NaCl and Pb(NO$_3$)$_2$ determined at room temperature are listed in Table 1 along with the values found with the Debye–Scherrer camera. For comparison purposes the lattice param-

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**Table 1. Comparison of the lattice parameter of some standard substances obtained with the focusing camera and a Debye–Scherrer camera, together with literature values at room temperature**

<table>
<thead>
<tr>
<th>Substance</th>
<th>Temperature ($^\circ$C)</th>
<th>Debye–Scherrer camera</th>
<th>Focusing camera</th>
<th>Literature values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>28</td>
<td>4.0498 ± 0.0002</td>
<td>4.0499 ± 0.0002</td>
<td>4.0499$^a$</td>
</tr>
<tr>
<td>Tungsten</td>
<td>25</td>
<td>3.1651 ± 0.0002</td>
<td>3.1651 ± 0.0002</td>
<td>3.1652±0.00009$^a$</td>
</tr>
<tr>
<td>Sodium chloride</td>
<td>25</td>
<td>5.6402 ± 0.0002</td>
<td>5.6404 ± 0.0002</td>
<td>5.6409±0.0005$^a$</td>
</tr>
<tr>
<td>Lead nitrate</td>
<td>25</td>
<td>7.8596 ± 0.0002</td>
<td>7.8595 ± 0.0002</td>
<td>7.8590±0.0009$^a$</td>
</tr>
</tbody>
</table>
The high-temperature calibration of the camera was performed by the measurement of the lattice parameter of Al at various temperatures and these values are listed in Table 2 along with the values of Wilson (1942). The \( a \) values of Al found here are in excellent agreement with those of Wilson.

The authors have investigated the isomorphic nitrates of lead, barium and strontium and have found that this camera possesses better resolution and precision than that of a Debye–Scherrer camera of the same radius. As an illustrative example we report here the accurate determination of the lattice parameter of \( \text{Pb(NO}_3\text{)}_2 \) between 25 and 275°C (Table 3).

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**References**


