Nauwkeurige bepaling der roosterconstanten van enkele mineralen bij hoge temperaturen.
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SUMMARY

This thesis deals with the accurate determination of the lattice constants of minerals at high temperatures. For this purpose a commercial 19 cm high-temperature powder camera was adapted to precision measurements. The minerals fluorite and fluorapatite were investigated up to 700°C and the results compared with the values deduced from powder photographs of the synthetic products.

In building up the apparatus (chapter I) special attention was paid to the stabilisation of the heater currents in order to maintain a constant temperature of the sample during the exposures. Moreover measurement of the pressure in the vacuum chamber proved to be necessary, since at pressures from 0.001 - 10 mm Hg the temperature of the furnace varies with the vacuum.

The inspection and the revision of the camera are described in chapter II. It was possible to measure the eccentricity as well as the angle between the axis of rotation and the axis of the film cylinder in a simple manner and to make them negligible by making the support of the specimen adjustable.

The calibration of the camera is discussed in chapter III. A simple method, requiring neither special apparatus nor the use of a calibrating substance, was developed for calibration of 19 cm cameras by film measurement (fig. 3.5).

Chapter IV deals with details of the experimental method and a discussion about the extrapolation procedures. For fluorite the graphical method after Taylor and Sinclair was chosen; for fluorapatite Cohen's least-squares method was preferred.

In order to control the absolute accuracy of the camera the lattice constants of aluminium and of Brazilian quartz were determined at 25.0°C. The results obtained are in good agreement with those of other authors as the following table shows.

<table>
<thead>
<tr>
<th>Lattice Constants at 25.0°C in Å</th>
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</thead>
<tbody>
<tr>
<td>( a_0 )</td>
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<tr>
<td>aluminium:</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
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<tr>
<td></td>
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<tr>
<td>brazilian quartz:</td>
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</tbody>
</table>
The transition point of quartz was used to test the reliability of the temperature indicator. Powder photographs show that the \( \alpha \) - and \( \beta \)-modifications coexist at this temperature (plate I, fig. 5.3) and that the transition is discontinuous, since the diffraction angles show a sudden change.

From the investigation of fluorite of Brienz (chapter VI) it appears that the thermal expansion is approximately linear up to 700\(^\circ\)C (fig. 6.1). The average linear coefficient of thermal expansion over the range 25 - 700\(^\circ\)C was found to be \((24.2 \pm 0.2) \times 10^{-6}\). There were no significant differences between the lattice constants of two minerals and a synthetic sample. The table below gives the cell dimensions at 25.0\(^\circ\)C (after heating).

<table>
<thead>
<tr>
<th>Mineral Type</th>
<th>( a_0 ) (Å)</th>
<th>( c_0 ) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluorite of Brienz</td>
<td>5.46339 ± 0.00010</td>
<td></td>
</tr>
<tr>
<td>Fluorite of Derbyshire</td>
<td>5.46349 ± 0.00021</td>
<td></td>
</tr>
<tr>
<td>Synthetic fluorite</td>
<td>5.46353 ± 0.00011</td>
<td></td>
</tr>
</tbody>
</table>

After heating, the parameters seem to be slightly smaller than before; for a definite conclusion more powder photographs are necessary.

About 200 reflections of fluorapatite were indexed (table 7.1). The difference in \( c_0 \) between the mineral and the synthetic specimen proved to be significant as is indicated by the following results at 25.0\(^\circ\)C:

\[
\begin{align*}
 a_0 & \text{ in Å} \\
 c_0 & \text{ in Å} \\
\text{Synthetic fluorapatite} & 9.37142 \pm 0.00027 \\
\text{Mineral fluorapatite} & 9.37111 \pm 0.00024
\end{align*}
\]

The difference is probably due to a small excess of calcium-phosphate in the synthetic product.

Both in the \( a \)- and the \( c \)-direction the thermal expansion of fluorapatite is nearly linear (fig. 7.1 and 7.2). Evaluation of the average coefficients of thermal expansion from 25 - 700\(^\circ\)C led to:

\[
\begin{align*}
 &a: (13.2 \pm 0.1) \times 10^{-6} \\
 &c: (12.7 \pm 0.2) \times 10^{-6}
\end{align*}
\]

On heating, the axial ratio remains nearly constant, diminishing less than 0.006\% per 100\(^\circ\).

The investigations both of fluorite and fluorapatite justify the conclusion that only extensive research can lead to determination of eventual minor deviations from the linearity of expansion.

LIT

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