values are ascribed correctly and it is desirable that approximate trial parameters are used. However, the program will work, with re-cycling, with any trial parameters. In practice it is usually found that two cycles of the program are necessary, excluding low-angle lines from the second cycle and possibly with the intermediate use of program (2). If only lines at high-angles can be used in the final cycle, the criticism that equal weight is given to all lines of the pattern in this method (Peiser, Rooksby & Wilson, 1960) is to some extent avoided. The output Table contains sufficient information for the application of de Wolff’s (1968) criterion for the reliability of powder pattern indexing. The operational time is usually between one and two minutes and 11000 core locations are used on the English Electric KDF9 computer.

References

A computer program for inverse pole figure statistics. By W. Dubroff, Inland Steel Research Laboratories, East Chicago, Indiana 46312, U.S.A.

(Received 30 November 1970; accepted 20 April 1971)

The relative volume fraction formulation of Horta, Roberts & Wilson (1969) is currently used in our inverse pole figure studies and is given below.

\[ f(h,k,l) = \frac{N(h,k,l)}{I_0(h,k,l)} \prod_{i=1}^{N(h,k,l)} \left( \frac{N(h,k,l)}{I_0(h,k,l)} \right)_i \]  
(1)

where

\[ f(h,k,l) \] = relative volume fraction of \{h,k,l\}.
\[ N(h,k,l) \] = multiplicity factor of \{h,k,l\}.
\[ I(h,k,l) \] = measured integrated intensity of \{h,k,l\} of sample.
\[ I_0(h,k,l) \] = measured integrated intensity of \{h,k,l\} of random sample.
\[ n = \text{number of measured } \{h,k,l\} \text{ diffraction maxima.} \]

Often the object of these investigations is to provide a quantitative interpretation of texture-dependent physical properties. Under these conditions, a quantitative characterization of the orientations present and the associated uncertainties is desired in order to eliminate erroneous applications and interpretations of the results.

The standard and fractional standard deviations of the relative volume fraction, \( f(h,k,l) \), and the intensity ratio, \( p(h,k,l) \), (Horta, Roberts & Wilson, 1969) have been derived from the theory of the propagation of errors under the assumption of time independent conditions (Beers, 1958). The results are extremely unwieldy and hence are best calculated by means of a computer. The standard deviation of \( f(h,k,l) \), \( \sigma_f \), is found to be

\[
\sigma_f = \left[ \frac{\left( \sum_{i=1}^{n} \left( \frac{N(h,k,l)_i}{I_0(h,k,l)} \sigma_{N(h,k,l)_i} \right) \right)^2}{\left( \sum_{i=1}^{n} \left( \frac{N(h,k,l)}{I_0(h,k,l)} \right)_i \right)^2} \right]^{1/2} + \left[ \sum_{i=1}^{n} \left( \frac{N(h,k,l)_i}{I_0(h,k,l)} \right) \right]^{1/2} \left[ \frac{\left( \sum_{i=1}^{n} \left( \frac{N(h,k,l)_i}{I_0(h,k,l)} \sigma_{N(h,k,l)_i} \right) \right)^2}{\left( \sum_{i=1}^{n} \left( \frac{N(h,k,l)}{I_0(h,k,l)} \right)_i \right)^2} \right] \]
\]

where \( \sigma_f \) and \( \sigma_{N(h,k,l)_i} \) are the standard deviations of \( I(h,k,l) \) and \( I_0(h,k,l) \), respectively.

It is important to note that \( \sigma_f \) can be reduced if \( \sigma_f \) and \( \sigma_{N(h,k,l)_i} \) are reduced. In most instances, the strength of the approach outlined above lies in the fact that confidence limits for a particular measurement can be evaluated on the basis of one measurement, instead of the usual practice which involves repeated measurements. Under these circumstances, it is clear that \( \sigma_f \) cannot be reduced, but that \( \sigma_f \) can be significantly reduced if we utilize the fact that the data are time independent. This can be accomplished by substituting the standard deviation of the average of several random samples, \( \sigma_{I_0} \), for \( \sigma_{I_0} \) in equation (2). \( \sigma_{I_0} \) will be significantly smaller than \( \sigma_{I_0} \) and can be shown to be (Beers, 1958):

\[ \sigma_{I_0} = \left[ q^{-2} \sum_{i=1}^{q} \sigma_{I_0}^2 \right]^{1/2} \]

(3)

where

\[ \sigma_{I_0} = \text{standard deviation of } I_0(h,k,l) \text{ for the average of random samples.} \]
\[ (\sigma_{I_0}) = \text{standard deviation of } I_0(h,k,l) \text{ for ith random sample.} \]
\[ q = \text{number of random samples.} \]

Improvements approaching a factor of at least five can be achieved by this averaging process.

A computer program has been developed for calculating \( f(h,k,l) \), \( p(h,k,l) \) and their respective statistical measures. Input for the program consists of the following:

1. Miller indices of each measured \{h,k,l\} reflection.
2. $2\theta$ scanning range and speed for each measured \(\{h,k,l\}\) reflection.
3. Correction factors for the separation of superimposed diffraction maxima.
4. \(I_0\) and \(\sigma_0\) values for the average of the random samples.
5. Total counts of the diffraction peak and background.

For a given set of experiments, provisions are made for storing items 1, 2, 3, and 4. At the present time, the program is being used for iron samples. Correction factors are available for the separation of the (411-330) and (442-600) diffraction maxima if the backgrounds are measured at a $2\theta$ position which is at least four to five half widths on either side of a particular diffraction maximum in order to minimize the amount of the peak's tail which is not recorded (Cohen, 1966). The program is written in basic Fortran IV and is currently used on an IBM 1800 under an MPX operating system. The program and details of the above derivations are available upon written request.

References


Crystal Data


New crystal data on dioxygenyl tetrafluoroborate, \(\text{O}_2\text{BF}_4\). By J. NORTON WILSON, RICHARD M. CURTIS and CHARLES T. GOETSCHEL,* Shell Development Company, Emeryville, California 94608, U.S.A.

*(Received 8 September 1970; accepted 8 December 1970)*

Origin of specimens

Samples were prepared by the reaction between boron trifluoride (BF\(_3\)) and oxygen difluoride (O\(_2\)F\(_2\)) as described previously (Solomon, Brabets, Venishi, Keith & McDonough, 1964; Keith, Solomon, Sheft & Hyman, 1968; Solomon, Keith, Kacmarek & Raney, 1968; Goetschel, Campanile, Wagner & Wilson, 1969) and were sealed under nitrogen in thin walled glass capillaries for X-ray photography.

* Present address: Kaiser Aluminum & Chemical Corporation, Center for Technology, P.O. Box 870, Pleasanton, California 94566, U.S.A.

Crystal geometry

Powder data were obtained with a Debye–Scherrer camera using Cu Ka radiation (1.5418 Ä). Intensities and positions were obtained from densitometer measurements of the films. The pattern was indexed on the basis of its similarity to NOBF\(_4\) (Evans, Rinn, Kuhn & Olah, 1964) and the assumption of the same space group is supported by both the required extinctions and the evidence noted below. With respect to extinctions only four ambiguities occur where allowed reflections are observed close to the positions of forbidden reflections. These are 220 near 003 (2.345 Å), 303 near 130 (1.820 Å), 031 near 023 (1.796 Å), and 421 near 510 (1.675 Å).

<table>
<thead>
<tr>
<th>(d_{obs})</th>
<th>(d_{cal})</th>
<th>(hk)</th>
<th>(\lambda)</th>
<th>(I/\lambda_0)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.50</td>
<td>5.490</td>
<td>101</td>
<td>20</td>
<td>2.189</td>
</tr>
<tr>
<td>4.38</td>
<td>4.389</td>
<td>200</td>
<td>10</td>
<td>2.162</td>
</tr>
<tr>
<td>3.90</td>
<td>3.914</td>
<td>011</td>
<td>34</td>
<td>2.122</td>
</tr>
<tr>
<td>3.73</td>
<td>3.724</td>
<td>111</td>
<td>6</td>
<td>2.099</td>
</tr>
<tr>
<td>3.53</td>
<td>3.518</td>
<td>201</td>
<td>11</td>
<td>2.095</td>
</tr>
<tr>
<td>3.45</td>
<td>3.450</td>
<td>002</td>
<td>26</td>
<td>2.095</td>
</tr>
<tr>
<td>3.26</td>
<td>3.265</td>
<td>210</td>
<td>100</td>
<td>2.095</td>
</tr>
<tr>
<td>3.10</td>
<td>3.098</td>
<td>211</td>
<td>16</td>
<td>2.095</td>
</tr>
<tr>
<td>2.820</td>
<td>2.819</td>
<td>112</td>
<td>85</td>
<td>2.043</td>
</tr>
<tr>
<td>2.736</td>
<td>2.791</td>
<td>020</td>
<td>74</td>
<td>1.955</td>
</tr>
<tr>
<td>2.702</td>
<td>2.745</td>
<td>202</td>
<td>11</td>
<td>1.937</td>
</tr>
<tr>
<td>2.486</td>
<td>2.488</td>
<td>121</td>
<td>46</td>
<td>1.937</td>
</tr>
<tr>
<td>2.434</td>
<td>2.432</td>
<td>212</td>
<td>6</td>
<td>1.937</td>
</tr>
<tr>
<td>2.354</td>
<td>2.355</td>
<td>311</td>
<td>6</td>
<td>1.937</td>
</tr>
<tr>
<td>2.266</td>
<td>2.266</td>
<td>320</td>
<td>8</td>
<td>1.937</td>
</tr>
<tr>
<td>2.243</td>
<td>2.250</td>
<td>221</td>
<td>61</td>
<td>1.937</td>
</tr>
</tbody>
</table>

Table 1. Powder data