Crystal Data

Crystal data for diphenylsilanediol, (C₆H₅)₂Si(OH)₂. By T. J. Kistenmacher, M. Rossi and L. K. Frevel, Department of Chemistry, The Johns Hopkins University, Baltimore, Maryland 21218, USA

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The cell constants of recrystallized diphenylsilanediol were refined from precision powder data taken with Cr Kα₁ (2.28962 Å): a = 14.493 (5); b = 15.012 (6); c = 9.897 (6) Å; α = 100.84 (9); β = 100.01 (5); γ = 120.77 (5); V = 1721.6 Å³;

Z = 6; Dₓ (25 °C) = 1.255 (3); Dₓ = 1.252 g cm⁻³.

Introduction

To resolve the conflicting unit-cell data for (C₆H₅)₂Si(OH)₂ (Kantz & Desando, 1968, versus Fawcett, Camerman & Camerman, 1977), specimens of recrystallized diphenylsilanediol were examined by single-crystal methods and by precision powder techniques.

Single-crystal data

Table 1 gives the cell constants obtained by different investigators. To conform to the standard convention for triclinic crystals (b > a > c), the labeling of a and c of the cell reported by Fawcett et al. (1977) has been interchanged. Except for c and γ, the cell constants reported by Kantz & Desando (1968) differ grossly from the data of the other three investigators and do not account for the observed powder pattern. If one changes α KD = 119.37° to 180° - α KD = 60.63° and then employs the transformation

\[
\begin{pmatrix}
a \\
b \\
c
\end{pmatrix}
\rightarrow
\begin{pmatrix}
0 & 1 & 0 \\
1 & 0 & 1 \\
0 & 0 & 1
\end{pmatrix}
\begin{pmatrix}
a \\
b \\
c
\end{pmatrix}
\]

one obtains a more compatible cell; namely, a = 14.36, b = 15.31, c = 11.0-98 Å, α = 101.73, β = 97.91, γ = 122.19°. From the interaxial angles measured by Kipping & Robison (1914), one can identify the interaxial angles of the reciprocal cell of the Deltauny reduced cell, namely, α* = 110:001 = 70.92°, β* = 110:001 = 71.32°, and γ* = 110:110 = 55.67°; hence α = 100.78, β = 100.03, γ = 120.86°.

Powder data

To obtain precision powder data, crystals of (C₆H₅)₂Si(OH)₂ were finely chopped with a razor blade and mixed with 9% by weight of cubic As₂O₃ (a = 11.0743 Å). The mixed powder specimen was loaded into a Hågg focusing camera (2r = 100 mm) equipped with a sample spinner. Crystal-monochromatized Cr Kα₁ radiation (2.28962 Å) was used to obtain sharp well resolved reflections. Dr. J. W. Edmonds of the Dow Chemical Company measured the films on an automated Jarrell-Ash microdensitometer coupled with a dedicated computer for data reduction. Columns 3 and 4 of Table 2 list the resultant averaged data from two separate films. Using 24 unambiguously indexed reflections in a least-squares refinement (Evans, Appleman & Handwerker, 1963), one obtains the unit-cell data quoted in Table 2. Columns 1 and 2 of Table 2 list all the permissible reflections. To conserve space the nil reflections are omitted after the 221 reflection.

To guard against any systematic errors in determining the axial lengths by single-crystal diffractometry, it appears advisable to utilize precision powder data taken with Cr Kα₁ and an internal standard to obtain a reference set of lattice constants.

The authors are indebted to the Dow Corning Corporation for samples of recrystallized diphenylsilanediol.

References


<table>
<thead>
<tr>
<th>Method</th>
<th>Density (g cm⁻³)</th>
<th>Reference</th>
</tr>
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<tbody>
<tr>
<td>4(Cu Kα₁)</td>
<td>1.25</td>
<td>Kantz &amp; Desando</td>
</tr>
<tr>
<td>Precision Weissenberg</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weissenberg, precession</td>
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<td></td>
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<tr>
<td>Cu Kα₁</td>
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<tr>
<td>Four-circle diffractometer</td>
<td>1.54178 Å</td>
<td>Fawcett et al. (1977)</td>
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<tr>
<td>Syntex goniometer</td>
<td>1.255 (25 °C)</td>
<td>Present work</td>
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<tr>
<td>Neutral buoyancy</td>
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<tr>
<td>Optical two-circle goniometer</td>
<td>1.54184 Å</td>
<td>Kipping &amp; Robison (1914)</td>
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</table>

Table 1. Single-crystal data on diphenylsilanediol
to several materials, including Gertner, Williams, Clarke, Pasko & Longo, 1976; Cheung, Andrews, Scherrer powder photographs.

Practice misfit causes a significant curvature of lattice parameters of epitaxic layers involving lattice misfit can be overcome by the use of the method described by Gandolfi (1967) for obtaining X-ray diffraction powder patterns from single-crystal specimens. The problems were: (1) lattice misfit causes a significant curvature of the substrate/layer structure which broadens the maxima in single-crystal diffractometer data excessively and thereby severely limits the precision attainable, and (2) the small mass of the epitaxic layers relative to that of the substrate and line broadening resulting from strains induced by crushing the specimens cause problems of sensitivity and resolution in powder diffractometer data and Debye-Scherrer powder photographs.

We have applied the Gandolfi method to several materials, including InAs, InSb/InAs (Andrews, Cheung, Gertner & Longo, 1976; Cheung, Andrews, Gertner, Williams, Clarke, Pasko & Longo, 1977) and Ga, InSb/GaSb (Rode, Gertner, Andrews, Cheung & Tennant, 1978). A sliver <1 mm wide, cleaved from the wafer, is a suitable specimen for the Gandolfi camera. To alleviate partially the lattice misfit in these materials, typical structures have as many as seven intermediate layers, which vary stepwise in composition and form a buffer region between the substrate and outermost active layers. The Gandolfi photographs show that, because of the relative layer thicknesses and X-ray absorption effects, the maxima of the substrate and outermost layers are strongly enhanced and clearly resolved from those of intermediate layers. This is very useful because the outermost active layers are of greatest interest and the substrate data provide standardization.

For the materials mentioned, there is a nearly linear relation between the cubic lattice parameter a and the compositional parameter x. Differences in their lattice parameters as small as 0.1% are easily resolved, which permits determination of the compositional parameter to the nearest percent. With greater care, an order of magnitude better precision, comparable to that attainable in conventional Debye-Scherrer photography, is probably possible. Minimizing the specimen size in the camera are both very important for attaining high precision.

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References


Table 2. Powder diffraction data of diphenylsilanediol

<table>
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<tr>
<th>hkl</th>
<th>d (Å)</th>
<th>d (Å)</th>
<th>10^2 I/I₀</th>
<th>hkl</th>
<th>d (Å)</th>
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<th>10^2 I/I₀</th>
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<td>10</td>
<td>002</td>
<td>5.12</td>
<td>5.12</td>
<td>10</td>
</tr>
</tbody>
</table>

*Note: The table contains data for powder diffraction patterns, with hkl indices and corresponding d-spacing values. The data is used for indexing and identifying crystal structures.*