Table 2. Density determinations on thallous formate saturated solutions*

<table>
<thead>
<tr>
<th>Method of determination†</th>
<th>( \rho ) (g.cm(^{-3}))</th>
<th>( t ) °C†</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.4813 (2)</td>
<td>24.46</td>
</tr>
<tr>
<td>B</td>
<td>3.40 (9)</td>
<td>22.26</td>
</tr>
<tr>
<td>B</td>
<td>3.42 (7)</td>
<td>22.96</td>
</tr>
<tr>
<td>B</td>
<td>3.42 (6)</td>
<td>22.66</td>
</tr>
<tr>
<td>B</td>
<td>3.44 (3)</td>
<td>23.46</td>
</tr>
<tr>
<td>C</td>
<td>3.43 (9)</td>
<td>23.46</td>
</tr>
<tr>
<td>C</td>
<td>3.43 (5)</td>
<td>21.35</td>
</tr>
<tr>
<td>C</td>
<td>3.41 (5)</td>
<td>22.66</td>
</tr>
<tr>
<td>C</td>
<td>3.45 (3)</td>
<td>23.56</td>
</tr>
<tr>
<td>C</td>
<td>3.45 (5)</td>
<td>23.66</td>
</tr>
<tr>
<td>C</td>
<td>3.46 (5)</td>
<td>23.96</td>
</tr>
<tr>
<td>C</td>
<td>3.46 (9)</td>
<td>23.96</td>
</tr>
<tr>
<td>C</td>
<td>3.45 (5)</td>
<td>23.96</td>
</tr>
<tr>
<td>D</td>
<td>3.48 (1)</td>
<td>24.46</td>
</tr>
<tr>
<td>D</td>
<td>3.47 (5)</td>
<td>24.46</td>
</tr>
</tbody>
</table>

† A - Pycnometer method (50 ml).
B - Germanium-Mylar composite flotation method.
C - Germanium-aluminum composite flotation method.
D - Archimedes method, using 90 g lump of intrinsic Ge.
† Temperature measurements made using 'Irident Thermometer' calibrated to ±0.01 °C.

and for the density of the water at 24.50°C. The value \( \rho_{\text{Ge}} = 5.322(3) \text{ g.cm}^{-3} \) was in good agreement with the X-ray density, which is ultimately dependent on the value taken for the atomic weight of Ge on the chemical scale (taken as 72.60).

When both the Ge and TiO composites were simultaneously in the hovering state, the density of the saturated thallous formate solution could then be computed from the Ge sample after which that of TiO could be obtained, without recourse to the Gmelin Table.

As a check on the modified flotation method, the density of the TiO sample was determined pycnometrically and by the Archimedes method of weighing in air and in water. The results were almost identical as shown in Table 1.

A complete set of density, lattice parameter and percent vacancies as a function of applied pressure and temperature will be published elsewhere.

It will be noted that the Ge composite flotation specimen yields the density of the saturated thallous formate solution as a function of the temperature at which the experiment is carried out. As an independent check on these values, the density of the solution was determined directly by means of a calibrated 50 ml. pycnometer at 24.46°C, and by means of Archimedes method using the same 89.5 gram single crystal of Ge as the standard. The results are listed in Table 2.

The experimental results are also shown in Fig.1 along with a portion of the Clerici curve for the same temperature range for comparison purposes. If our pycnometer value of 3.4813(2) at 24.46°C is taken as the standard value, the density over the range 20–28°C approximately can be written in the form

\[ \rho_{\text{th}} = 3.4813(2) + 0.0342(t - 24.46) \text{ g.cm}^{-3} \]

References

creases strongly with increasing distance from the circle, due to the decrease of the atomic scattering factor for electrons and to the influence of slight deviations from the ideal orientation. Taking this into account the theoretical and the experimental intensity distributions match very well.

Closer inspection of the photographs shows an additional feature: diffuse rings which appear to start from the tangential streaks and fade into the area of non-zero intensity of the interference function. A detailed study of this effect reveals that these diffuse rings are due to a modulation of the \((hk)\) rods indicating a beginning of graphitization in these particular fibres. (All fibres with lower preferred orientation studied so far

![Fig. 1. Electron diffraction photographs of a highly oriented carbon fibre.](image1)

**Experimental**

The electron diffraction studies were carried out in a Siemens electron microscope (Elmiskop 1) at 100 kV on fragments of powdered fibres. The fibres were made available by Dr R. Bacon (Union Carbide Corporation, Carbon Products Division).

**Results and discussion**

Typical photographs are given in Fig.1. In Fig.2 an intensity map of the theoretical interference function of an \((hk)\) interference for a perfectly oriented carbon fibre \((q = -1)\) with infinite and perfect layers is given. This corresponds to

\[
I(s, \varphi) = \frac{1}{\pi^2 |(s^2 - s_0^2)(s_a^2 - s^2 \cos^2 \varphi)|}
\]

according to the general treatment given in the earlier paper (Ruland & Tompa, 1968; the parameters used are the same as in this paper).

When comparing the two figures one has to keep in mind that the intensity of the tangential streaks de-

![Fig. 2. Theoretical intensity distribution of an \((hk)\) interference for perfect orientation of the layer planes parallel to the principal axis.](image2)

![Fig. 3. Schematic presentation of the truncation of 10l interferences due to high preferred orientation.](image3)
the principal axis of the fibre fragments is not perpendicular to the electron beam so that the plane section of reciprocal space as given by the photograph does not contain this axis. Fig. 5 shows a schematic presentation of such a section. In Fig. 5(a) the contours of \( I(s, \phi) \) for prefect orientation in a plane containing the fibre axis are given, together with the angle \( \alpha \) between the fibre axis and an oblique section. The contours of \( I(s, \phi) \) on the oblique section are given in Fig. 5(b). From this, one can deduce that the ratio of the diameter of the inner contour to the distance between the outer contours gives the cosine of \( \alpha \) and thus a measure of the inclination of the fibre axis to the electron beam. Furthermore, since the outer contours determine the truncation of the interference rings with non-zero \( l \), all those with a radius smaller than \( s_h / \cos \alpha \) will not be truncated. With this, one obtains \( \alpha = 22^\circ \) and \( \alpha = 35^\circ \) for the two photographs shown in Fig. 4, from which one calculates that the truncation starts with the interference rings of \((102), (114), (203), (214), \text{etc.}\) and \((103), (114), (205), (216), \text{etc.}\) respectively, which is in accordance with the observation.

A complete quantitative interpretation taking into account the effect of imperfect orientation, finite size and perfection of the layers and the type of interlayer ordering can be obtained following the development given in section 2.6 of the earlier paper (Ruland & Tompa, 1968). Such studies are under way.

References
