TEM Practicum Exercise – indexing selected area electron diffraction pattern (#5)
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1 Introduction

Historically, Transmission Electron Microscopes were developed mainly due to the limited image resolution of light microscopes, which are restricted to the visible light wavelengths. Today, the TE microscopes are undoubtedly the most efficient way of characterizing a material, especially for its electron diffraction measurement which can provide information on the crystal structure, lattice repeat distance and specimen shape, among other things.

The TEM forms the images in two different modes:
- Imaging Mode
- Diffraction Mode or Selected Area Diffraction

1.1 Electron Diffraction Modes

The selected area electron diffraction (SAED) is a crystallographic experimental technique carried by the TEM in which the user can select an area of the specimen in which the diffraction pattern is desired. The selected area aperture – a thin strip of metal with different sized holes that blocks the beam - can be inserted below the sample holder and into the beam path.

For thin crystalline samples, a diffraction pattern can be an image of a pattern of dots in the case of a single crystal or a series of rings in the case of a polycrystalline material. For the single crystal, the diffraction pattern is dependent on the orientation of the specimen and the structure of the sample being illuminated by the electron beam.

The goal of this exercise is to index the images formed by the diffraction mode. Using the SAED pattern, one will measure the length of the reciprocal lattice vector g and the angle between observed reflections. Afterwards, d will be calculated to find the corresponding (h,k,l) values. Finally, the crystal zone axis is determined.
## Indexing a Selected Area Electron Diffraction Pattern SAEDP

![Selected area electron diffraction pattern (SAEDP) taken from aluminum alloy.](image)

**Figure 1:**

1. In order to analyse a SAEDP, firstly a parallelogram with the smallest $g_1$, $g_2$ and $g_3$ is chosen on the spot electron diffraction pattern.
   
   a. Measurement of the size of $g_1$, $g_2$ and $g_3$ and the scale bar is done with a ruler.
   
   b. The values are then compared with the actual size on the scale bar.
   
   c. The length $|g_i|$ in nm$^{-1}$ is determined using the scale bar ratio on the diffraction pattern;

   **Table 1:** $g_i$ was calculated from the relation of scale bar on a ration 6 cm $\rightarrow$ 15 nm$^{-1}$.

<table>
<thead>
<tr>
<th>Index $i$</th>
<th>$g_{hkl}$ nm$^{-1}$</th>
<th>$g_{hkl}$ cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.25</td>
<td>1.7</td>
</tr>
<tr>
<td>2</td>
<td>5.00</td>
<td>2.0</td>
</tr>
<tr>
<td>3</td>
<td>4.25</td>
<td>1.7</td>
</tr>
</tbody>
</table>

2. Afterwards, the inter planar distance $d_i$ is determined by the equation

   $$d_i = \frac{1}{|g_{hkl}|}$$
These values are then compared with the given table in the exercise:

Table 2: Determination of inter planar distances, d values, family planes and planes.

<table>
<thead>
<tr>
<th>Index i</th>
<th>$\overline{g}_{hkl}$ nm$^{-1}$</th>
<th>$d_{hkl}$ nm</th>
<th>Family plane</th>
<th>Plane</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.26</td>
<td>0.234</td>
<td>{111}</td>
<td>(111)</td>
</tr>
<tr>
<td>2</td>
<td>4.26</td>
<td>0.234</td>
<td>{111}</td>
<td>(\overline{1}\overline{1}1)</td>
</tr>
<tr>
<td>3</td>
<td>4.92</td>
<td>0.203</td>
<td>{002}</td>
<td>(002)</td>
</tr>
</tbody>
</table>

In order to find the plane (hkl), the following formula shall be applied:

\begin{align*}
h_1 + h_2 &= h_3 \\
k_1 + k_2 &= k_3 \\
l_1 + l_2 &= l_3
\end{align*}

Thus in \{hkl\}→{111}, {111} and {002} we have:

\begin{align*}
1 - 1 &= 0 \\
1 - 1 &= 0 \\
1 + 1 &= 2
\end{align*}

And the plane will then be the column of numbers with the appropriate signal chosen.

iii. In order to measure the angles, there are two possibilities:

a. The family planes are inserted in the formula

$$
\cos \varphi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\sqrt{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)}}
$$

b. Draw an angle (using the protractor template)
The values obtained were the following:

\[
\begin{align*}
\varphi_1 &= \cos^{-1} \frac{(h_1h_3+k_1k_3+l_1l_3)}{\sqrt{(h_1^2+k_1^2+l_1^2)(h_3^2+k_3^2+l_3^2)}} = \cos^{-1} \frac{0+0+2}{\sqrt{12}} = 54.7^\circ \\
\varphi_2 &= \cos^{-1} \frac{(h_2h_3 + k_2k_3 + l_2l_3)}{\sqrt{(h_2^2 + k_2^2 + l_2^2)(h_3^2 + k_3^2 + l_3^2)}} = \cos^{-1} \frac{0 + 0 + 2}{\sqrt{12}} = 54.7^\circ
\end{align*}
\]

| Table 3: Interplanar angles for cubic system |
|-------------------------------|----------------|
| \(\varphi_1\)                | \(\varphi_2\)  |
| Measurement                  | 52\(^\circ\)   | 56\(^\circ\) |
| Calculation                  | 54.7\(^\circ\) | 54.7\(^\circ\) |

The values correspond to each other and can be considered satisfactory.

On the second task, one must determine the crystal zone axis for the indexed diffraction spots (i.e. crystal planes in a real space). This can be obtained with the following formula:

\[
<uvw> = \{h_1k_1l_1\} \times \{h_2k_2l_2\}
\]

\[
<uvw> = (111)x(002) =
\begin{bmatrix}
i & j & k \\
i & 1 & 1 \\
1 & 0 & 1
\end{bmatrix} = <220>
\]

General Family of Directions

If we calculate the direction parallel to the electron beam specifically for one set of planes:

\[
<uvw> = (\overline{1}11)x(002) = [\overline{2}20]
\]

\[
\begin{bmatrix}
i & j & k \\
1 & 1 & 1 \\
1 & 0 & 2
\end{bmatrix} = -2i + 2j + 0k = [\overline{2}20] = \{\overline{1}10\}
\]

This is a particular solution for particular indices of planes, thus brackets should be square.

The zone axis is a direction in the crystal which is parallel to the electron beam. Thus this axis is [\(\overline{1}10\)]. Indices must be reduced to least numbers.
3 References
