ANOMALOUS SCATTERING FROM SINGLE CRYSTAL SUBSTRATE

L. K. Bekessy, N. A. Raftery, and S. Russell

Faculty of Science, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland, Australia

ABSTRACT

The nature of the scattering pattern for silicon 004 wafer is described. At incidence angles ($\omega$) set close to the expected diffraction condition of $\omega = \theta_{h'k'l'} - \alpha_{hk'k'l'}$ ($hkl$ are the planes parallel to the wafer surface and $\alpha_{hk'k'l'}$ is the interfacial angle between $hkl$ and another set of diffracting planes $h'k'l'$), an anomalous kind of scattering has been observed. There is a broad weak feature at about the expected diffraction angle of $2\theta_{h'k'l'}$ and a narrow feature whose position depends solely on the incidence angle. The broad feature is the Bragg diffraction of the X-ray tube spectral components while the narrow feature is a non-Bragg scattering peak. Both features can be eliminated in some cases with knowledge of the orientation of the wafer to the diffractometer axes.

INTRODUCTION

Silicon single crystal wafer and other single crystal materials are used as thin film substrate. The advantage of a single crystal wafer substrate is that while the wafer may diffract strongly in some directions, it scatters weakly in most (non-Bragg) directions. Glancing incidence (or shallow angle asymmetric parallel beam) X-ray diffraction is the prevalent geometry used when analysing thin film samples. The low incidence angle keeps the X-ray beam nearly parallel to the surface region and the diffraction intensity does not suffer greatly at higher diffraction angles as is the case for a symmetric geometry. The scattering pattern of the substrate is important in the determination of the nature of the thin film X-ray diffraction pattern.

A wafer is usually cut such that one set of planes ($hkl$) is parallel to the wafer surface. Since in glancing incidence the incidence angle ($\omega$) is set to a low value (a few degrees), the incidence angle would be well away from $\omega = \theta_{hkl}$ and the strong diffraction peak which would be expected at $2\theta_{hkl}$. There are, however, other sets of planes present which are at defined (interfacial) angles ($\alpha_{hk'k'l'}$). If $\alpha_{hk'k'l'} + \omega = \theta_{h'k'l'}$, then a strong diffraction peak would be expected at $2\theta_{h'k'l'}$.

At regions around the angles $2\theta_{h'k'l'}$ other effects may come into play depending on the relationship between $\alpha_{hk'k'l'}$ (interfacial angle), $\omega$ (incidence angle), $2\theta$, $\phi$ (rotation angle), and $\chi$ (orientation angle). The diffractometer angles are defined in Figure 1.
EXPERIMENTAL DETAILS

The equipment used was a PANalytical X’Pert Pro diffractometer with a PW3050/60 theta/theta goniometer and a PW3373/00 copper long fine focus X-ray tube. A PW1348/66 X-ray mirror was used to produce a parallel beam that is approximately 1.2 mm in height with 20 mm width. Soller slit packs of 0.04 rad are used in the pre and post diffraction optics. The post diffraction optic is a 0.09° parallel beam collimator with a large area (25 mm × 20 mm) proportional detector.

A low but varied incidence angle (ω) was used in the range 1 to 10°. This is well away from the Bragg angle for silicon θ_{004}.

The sample is commercial silicon wafer cut parallel to the (001) planes, approximately 40 mm × 40 mm × 0.8 mm. The surface was etched to minimize the oxide layer.

BACKGROUND

Anomalous scattering was observed emanating from a silicon substrate when thin powder layers were examined at low incidence angles. These scattering events occurred in the proximity of some silicon diffraction peaks. The nature of these effects was perplexing in that it could not be interpreted as Bragg diffraction. It was known that certain incidence angles are to be avoided as they would bring certain planes into the diffracting geometry (see Table I). It could be seen that there was a relationship between these key incidence angles and the anomalous behaviour. A pattern showing this behaviour can be seen in Figure 2. The diffraction pattern was obtained from the substrate only.
Table I. Interfacial angles and key incidence angles for silicon 004.

<table>
<thead>
<tr>
<th>$2\theta$(CuK$\alpha$)</th>
<th>hkl</th>
<th>Interfacial angle $\alpha$ for silicon 004</th>
<th>$\omega_i$ omega - interference</th>
</tr>
</thead>
<tbody>
<tr>
<td>28.44</td>
<td>111</td>
<td>54.74</td>
<td>~</td>
</tr>
<tr>
<td>47.3</td>
<td>220</td>
<td>45  90</td>
<td>~</td>
</tr>
<tr>
<td>56.12</td>
<td>311</td>
<td>25.24 72.45</td>
<td>2.82 ~</td>
</tr>
<tr>
<td>69.13</td>
<td>400</td>
<td>0  90</td>
<td>34.57 ~</td>
</tr>
<tr>
<td>76.38</td>
<td>331</td>
<td>46.51 76.74</td>
<td>~</td>
</tr>
<tr>
<td>88.03</td>
<td>422</td>
<td>35.26 65.91</td>
<td>8.76 ~</td>
</tr>
<tr>
<td>94.95</td>
<td>511</td>
<td>15.79 78.9</td>
<td>31.69 ~</td>
</tr>
<tr>
<td>106.72</td>
<td>440</td>
<td>45  90</td>
<td>8.36 ~</td>
</tr>
<tr>
<td>114.09</td>
<td>531</td>
<td>32.31 59.53</td>
<td>80.27 24.74 ~</td>
</tr>
<tr>
<td>127.54</td>
<td>620</td>
<td>18.43 71.45</td>
<td>90  45.34 ~</td>
</tr>
<tr>
<td>136.89</td>
<td>533</td>
<td>40.32 62.77</td>
<td>28.13 5.67 ~</td>
</tr>
</tbody>
</table>

Figure 2. Behaviour of silicon 004 substrate at incidence angle ($\omega$) of 4 deg.

The broad feature is at 56.06° and the narrow feature at 58.50°. These features, if present, may completely dominate the sometimes weak thin film pattern. The position and intensity of these peaks were found to vary from one thin film preparation to the next.

EXPLANATION OF BEHAVIOUR

In order to understand the above behaviour and to seek to avoid it if possible, it was decided to closely examine the scattering from the 004 planes of silicon. In Figure 3 there is a graphical display of this behaviour for a variation of the incidence angle of +/- 3 deg. ($\omega$) with respect to the symmetric case ($\omega = \theta_{004}$). The symmetric data is measured with a known attenuator and rescaled for comparison. It can be seen that the broad feature is moving only slightly with $\omega$ whilst the narrow feature moves dramatically but linearly with $\omega$. 
Figure 3. Behaviour of silicon 004 substrate scattering with respect to the variation of the incidence angle from the symmetric situation ($\omega = \theta_{004}$). $\omega = \theta_{004}$ (variation = 0 deg.) data rescaled, + 3 deg., + 1 deg., -1 deg., -3 deg.

The intensity of both features decreases as $\omega$ is rotated away from $\theta_{004}$, with the broad feature decreasing in intensity more quickly than the narrow feature.

An explanation of the behaviour is that the broad feature is the diffraction of the tube (source) wavelengths. It is greatest in the symmetric case ($\omega = \theta_{004}$) when $K\alpha$ is diffracting and falls off rapidly as $\omega$ is changed away from $\theta_{004}$ as non-characteristic radiation is diffracted.

An explanation of the behaviour for the narrow feature is not complete. Further experimentation and analysis is required. It is a non-Bragg scattering peak. This narrow feature is at its most intense closest to the symmetric case ($\omega = \theta_{004}$) and decreases rapidly as $\omega$ is changed from $\theta_{004}$. It is discernable above the background for up to 15° ($2\theta$) either side of the expected (Bragg) diffraction peak position.

In an effort to understand this behaviour, the {311} diffraction planes of the 004 silicon were investigated. The {311} planes have a strict crystallographic relationship to the {004} planes. From the perspective of the 004 planes, there is a four-fold symmetry for the {311} planes of which only four would be close to the Bragg condition for an incidence angle of 4° ($\omega$). It would be expected that a rotation in $\phi$ (see Figure 1) would displace these from a possible scattering condition. A rotation in $\phi$ resulted in a decrease in the intensity for the non-Bragg effect (see Figure 4). If the 004 silicon wafer were placed at an arbitrary orientation in $\phi$ then the peaks would occur 90°, 180°, and 270° ($\phi$) from the initial peak position—the higher $\phi$ data is not plotted in Figure 4.
CONCLUSIONS

The broad feature is the diffraction of the source wavelengths; its position changes slightly as the value of $\omega$ changes (see Figures 2 and 3).

The narrow side feature is due to a non-Bragg scattering for which the authors do not have a complete explanation; its position varies linearly with $\omega$ (see Figure 3).

The non-Bragg scattering can be seen against the generally low background of the silicon wafer up to +/- 15° ($2\theta$) depending on which planes are scattering.

Rotation in $\phi$ will cause interfacial planes to move out of the scattering situation in some cases (see Figures 1 and 4), but has no effect around $\omega = \theta_{004}$.

Care should be taken in selecting silicon wafer (or for that matter any single crystal substrate) cut direction (001, 511, etc.) and the orientation relative to the diffractometer axes. These two aspects determine the range of $2\theta$ where the substrate contribution can be ignored when this material is used as a thin film substrate in glancing incidence analysis.

ACKNOWLEDGEMENT

The authors would like to thank Professor Paul Fewster, PANalytical Research Centre for his help in interpreting the behaviour.