The \( \pi/2 \) Side-Reflection Laue Technique and the New Chart

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Abstract

For the capability of dynamic studies of structural changes of crystals under the environment of heat, electric or magnetic field, the \( \pi/2 \) side-reflection Laue technique is performed in which the X-ray source, the specimen and the film are aligned along an L-shaped track. A new chart has also been designed for the analysis of \( \pi/2 \) side-reflection Laue patterns. This new chart is applied to the analysis of crystal orientation in the \( \pi/2 \) side-reflection Laue technique and to indexing the planes of simultaneous multiple-reflection images in Berg-Barrett topography. Also, the equation of zonal trace has been derived for depicting the zonal curves of configurations of \( \pi/2 \) side-reflection spots and confirming the results which are analyzed by the new chart.

1. Introduction

When white radiation is used to illuminate a crystal, Laue diffraction is produced (Friedrich, Knipping & von Laue, 1912; Azaroff & Donahue, 1969; Amoros, Buerger & Canut de Amoros, 1975). This Laue technique is classified into both transmission and back-reflection methods. In the transmission arrangement the specimen is placed between the X-ray source and the film. With the back-reflection arrangement the film is placed between the X-ray source and the specimen. One way of determining the crystal plane orientation is by means of the Leonhardt chart for the transmission method (Dunn & Martin, 1949) and the Greninger chart for the back-reflection method (Greninger, 1935).

The development of this technique over the past two decades (Bordas, Munro & Glazer, 1976; Steinberger, Bordas & Kalman, 1977; Wood, Thompson & Mathewman, 1983; Hajdu et al., 1987; Harding, Maginn, Campbell, Clifton & Machin, 1988; Hajdu & Johnson, 1990) has been due to improvements in the production of white X-rays at synchrotron radiation sources and to developments in computing techniques for processing the data (Machin, 1985, 1987; Campbell, Habash, Moffat & Helliwell, 1986; Campbell et al., 1987; Rabinovich & Lourie, 1987; Helliwell et al., 1989; Shrive, Hajdu, Clifton & Greenhough, 1990; Clifton, Elder & Hajdu, 1991).

Also, various electro-optimal systems (hereinafter image detectors) have been reported in the early sixties which permit intensification of X-ray diffraction patterns and thus a decrease in exposure time for recording and displaying the X-ray images (Weyerer, 1961; Green, 1970, and references therein). Newly developed two-dimensional detectors such as position-sensitive multiwire detectors (Hamlin, 1985), TV detectors (Arndt, 1985) and imaging plates (Miyahara, Takahashi, Ame-miya, Kaniya & Satow, 1986) have substantially improved such defects of X-ray films as chemical fogs, narrow dynamic range, troublesome optical densitometry etc. Real-time observations of transmission Laue patterns by image detectors offer many advantages over conventional photographic methods. They provide a fast and convenient way for determining the orientation of single crystals and also offer the additional capability of dynamic studies of structural changes as a function of both orientation and time. However, transmission Laue experiments for real-time observations require thin specimens of low absorption. Furthermore, real-time observations of in situ transmission Laue experiments under the environment of heat, electric or magnetic field are no longer carried out because the installations for the environments are not free. If the electric field is applied perpendicular to a specimen wafer which is mounted parallel between flat electrodes, the electrodes cut off both incident and transmitted beams. If the electrodes make an angle of \( \pi/4 \) with the incident beam, however, the reflected patterns may be observed at a \( \pi/2 \) locus by either the film or the image detector. Therefore, we report a new \( \pi/2 \) side-reflection technique which aligns the X-ray source, specimen and film (or image detector) along an L-shaped track. Also, a new chart is constructed for the analysis of \( \pi/2 \) side-reflection patterns. The new chart can be used to index the diffraction planes of multiple-reflection patterns. It can also be used to index the diffraction planes of multiple reflections in Berg-Barrett topography (Turner, Vreeland & Pope, 1968).

Applications of the new chart have been performed for both the analysis of crystal orientation in the side-
reflection technique and the index of diffraction planes of multiple reflections in Berg–Barrett topography with a silicon wafer.

Also, the algebraic equation of zonal trace is derived which represents the zonal curves of configurations of \( \pi/2 \) side-reflection spots. This equation is used to depict the zonal traces for the additional confirmation of orientation analysis of crystals.

Finally, a \( \pi/2 \) side-reflection experiment has been performed using the image detector for the real-time observation of this experiment.

2. Side reflection

2.1. The new chart

In the \( \pi/2 \) side-reflection arrangement the X-ray source is placed at the north pole, \( N \), the specimen at the center, \( C \) and the film in the tangential plane of the point \( A \) on the octant of a reference sphere, as shown in Fig. 1. Therefore, the source, the specimen and the film are aligned in an \( L \) shape forming a right angle.

The X-rays from the source located at \( N \) are incident on the specimen crystal at \( C \), make an angle \( \chi \) with a crystal-plane normal, and are reflected by \( 2\chi \) in the optical plane \( NCMN \). Here, the crystal-plane normal vector, \( \overrightarrow{CP} = n_{hkl} \), is located at the angular position of latitude \( \psi \) and longitude \( \varphi \). Then,

\[
\begin{align*}
n_{hkl} &= in_x + jn_y + kn_z \\
n_x &= n_{hkl} \cos \psi \sin \varphi \\
n_y &= n_{hkl} \sin \psi \\
n_z &= n_{hkl} \cos \psi \cos \varphi
\end{align*}
\]

and

\[
\begin{align*}
\sin \psi &= n_x/n_z \\
\tan \varphi &= n_x/n_z \\
\end{align*}
\]

Let us consider the unit vector of the scattered X-ray beam,

\[
u_{hkl} = u_x + jv_y + ku_z.
\]

Then, the diffraction vector \( S_{hkl} \) becomes

\[
S_{hkl} = ix + jy + kx = |S_{hkl}|u.
\]

From Fig. 1, which shows the position of an X-ray diffraction spot \( S \) scattered from a crystal, the \( x \) position of the spot \( S \), \( x \), coincides with the crystal-to-film distance, \( D \),

\[
x = D = |S_{hkl}|u_x.
\]

So,

\[
|S_{hkl}| = x/u_x.
\]

From (3)–(6)

\[
z = xu_x/u_x = x \tan \varphi
\]

and

\[
y = xu_y/u_x = x \cot 2\chi \sec \varphi.
\]

If the angle \( \chi \) is a constant, the algebraic equation relating (7) to (8) becomes a hyperbolic,

\[
y^2 \tan^2 2\chi - z^2 = x^2.
\]

Using (7) and (9) we can make a new chart which represents the angular positions of diffraction spots in the \( \pi/2 \) side-reflection technique, as shown in Fig. 2. This chart is for \( D = 3 \text{cm} \), \(-60 < \varphi < 60^\circ \) and \( 15 < \chi < 75^\circ \).

2.2. The equation of zonal trace

The zonal trace of back-reflection Laue spots is a hyperbola consisting of a Greninger net. According to Yeom, Yoon & Park (1994) the equation of zonal trace is expressed as

\[
\left( \frac{\sin^2 \varphi_{uvw}}{B^2} - \frac{\cos^2 \varphi_{uvw}}{A^2} \right)x^2 + \left( \frac{\cos^2 \varphi_{uvw}}{B^2} - \frac{\sin^2 \varphi_{uvw}}{A^2} \right)z^2 - \sin 2\varphi_{uvw} \left( \frac{1}{B^2} + \frac{1}{A^2} \right)xz = 1,
\]

where \( A \) and \( B \) are parameters related to the crystal structure.

Fig. 1. Schematic diagram of the octant of a reference sphere showing the alignment of the \( \pi/2 \) side-reflection. \( N \): position of X-ray source; \( C \): a crystal; \( A \): the tangential point of the octant with a flat film parallel to the \( yz \) plane; \( \overrightarrow{CP} \): a plane-normal vector in a crystal (or a reciprocal-lattice vector \( n_{hkl} \)); \( \psi \): the latitude of a point \( P \); \( \varphi \): the longitude of a point \( P \); \( \pi/2 - \psi \).
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where

$$A^2 = \frac{4y^2}{(2 - 3 \tan^2 \gamma_{hkl})},$$

$$B^2 = y^2 \tan^2 2\gamma_{hkl},$$

and $\varphi_{uvw}$ is the angle between the projected zone axis on the $xz$ plane and the $z$ axis (vertical), $\gamma_{hkl}$ is the tilt angle of the zone axis from the vertical plane, and $y$ is the crystal-to-film distance. Also, the intersecting line of the hyperbolic surface (10) and the vertical plane which is $x = D$ is expressed as follows:

$$y^2 = \frac{\cos^2 \varphi_{uvw} - \frac{(2 - 3 \tan^2 \gamma_{hkl})}{4} \sin^2 \varphi_{uvw}}{\tan^2 \gamma_{hkl}} z^2 + D \sin 2\varphi_{uvw} \left[ \frac{1}{\tan^2 2\gamma_{hkl}} + \frac{(2 - 3 \tan^2 \gamma_{hkl})}{4} \right] z^2$$

$$= \frac{\sin^2 \varphi_{uvw} - \frac{(2 - 3 \tan^2 \gamma_{hkl})}{4} \cos^2 \varphi_{uvw}}{\tan^2 2\gamma_{hkl}} D^2.$$  

Equation (11) represents the zonal trace in the $\pi/2$ side reflection.

3. Experiments

In the $\pi/2$ side-reflection technique the X-ray source–specimen alignment is perpendicular to the specimen–film alignment. Therefore, the X-ray source, the specimen and the film are aligned along an L-shaped track, i.e. the X-ray source is positioned at one end of the L, the film at the other end and the specimen at the bent corner. Confirming the accurate film centre and specimen-to-film distance is very important for the $\pi/2$ side-reflection analysis.

![Fig. 2](image-url)  
Fig. 2. The new chart for the $\pi/2$ side-reflection Laue method where $D = 3$ cm.

![Fig. 3](image-url)  
Fig. 3. (a) The Berg–Barrett topograph of a silicon wafer which has (001) crystal plane orientation. (b) the $\pi/2$ side-reflection corresponding to (a).
A π/2 side-reflection Laue photograph and a back-reflection Laue photograph of a silicon wafer, which has (001) crystal-plane orientation, were obtained using Cu radiation at 30 kV, 40 mA, 80 min exposure and 3 cm specimen-to-film distance for the confirmation of coincidence with the back-reflection technique.

Berg–Barrett topography of the silicon wafer, which has (001) crystal-plane orientation, was performed using CuKα radiation at 30 kV, 40 mA, 5 min exposure and 3 cm specimen-to-film distance. In this experiment two reflections in addition to the 004 reflection were obtained, as shown in Fig. 3(a). To index the two reflections another π/2 side-reflection experiment was performed, as shown in Fig. 3(b).

Finally, a real-time observation of a π/2 side-reflection was performed using Cu radiation at 40 kV and 100 mA and 1.5 cm specimen-to-detector distance with a Rigaku Rotaflex generator (18 kW) and a Bede 50 mm X-ray imager (Fig. 4).

4. Results and discussions

By comparing the crystal orientation analysis of a silicon wafer by the π/2 side-reflection geometry with that by the back-reflection technique we have confirmed that the π/2 side-reflection technique poses no problem for the analysis of crystal orientation. Another application of the new chart was to index the multiple-reflection planes, as shown in Fig. 3(a). The reflections are of the (004), (115) and (224) planes. Therefore, its utilization for indexing shortens the analyzing time and clarifies the analysis better than that of diffraction loci circles (Turner, Vreeland & Pope, 1968).

Usually, the Grenninger net is used to read the angular coordinates of zone axes as well as that of diffraction spots. However, the new chart can only be used to read the angular coordinates of diffraction spots. Of course, it is possible to find the angular coordinates of zone axes if we represent the loci of plane normals in a Wulff net which is read by means of the new chart.

Also, if we substitute the angular coordinates of the zone axis into (11) we can depict the zonal traces of the diffraction spots. We have shown the orientation analysis of Fig. 3(b) to be correct by comparing the depicted curves with the diffracted traces.

Finally, for the capability of dynamic studies of structural changes of crystals under the environment of heat, electric or magnetic field, we have performed a pre-experiment of a (001) Si wafer using Cu radiation at 40 kV, 100 mA, 1.5 cm specimen-to-image detector distance with a Rigaku Rotaflex generator and Bede 50 mm X-ray imager (Fig. 4). From this figure we believe the π/2 side-reflection would be positively utilized for the real-time observation of in situ Laue experiments under the environment of heat, electric or magnetic field.

Also, we anticipate the need for another side-reflection technique such as π/4, and others such as 3π/4, because of installation convenience for the environment. Then we can obtain solutions for constructing nets for all side reflections if we operate the transformation matrix for a rotation of the primed coordinate system through an angle ±0 rot about the z axis to the xyz system in equations (7) and (9).

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References


