Analysis of Kikuchi band contrast reversal in electron backscatter diffraction patterns of silicon

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1. Introduction

Electron backscatter diffraction (EBSD) is a powerful tool in the crystallographic characterization of materials [1–5]. In EBSD, the local resolution of the focussed electron beam in a scanning electron microscope is utilized to observe Kikuchi patterns of electrons backscattered from spatially confined sample regions. As electron beams can be focussed to nanometer dimensions and because the Kikuchi patterns are tied to the local crystallographic order, EBSD thus allows to investigate the microstructure of materials on comparable scales.

In spite of the conceptual simplicity of the experimental setup used in EBSD, in principle consisting only of a sensitive phosphor screen and a fast CCD camera, the actual formation process of EBSD patterns is complicated by the multiple elastic and inelastic scattering of the observed electrons inside the sample. It can be expected that an improved understanding of the physical effects relevant for EBSD pattern formation will allow us to better quantify the possibilities and limits of the EBSD technique and to extract additional information from experimental EBSD measurements by comparison to quantitative simulations. Such simulations are also important to identify experimental situations in which simplified models (e.g. kinematical, single-scattering approximations) can be used.

The fact that simple kinematical models are of limited use for the description of the actual intensity distribution observed in EBSD patterns is exemplified already in the early investigations of Alam et al. [6] who measured characteristic differences in the backscattered Kikuchi patterns when the angle of incidence of the primary electron beam on the sample was changed. A very striking observation was that in specific regions of the patterns, the Kikuchi bands turned from showing maximum intensity in the middle (bright excess bands) to a minimum of intensity (dark deficit bands). The evolution from bright to dark Kikuchi bands under a change of the incident beam geometry illustrates directly that, in order to describe EBSD patterns quantitatively, we need models that can correctly handle the physical processes behind the complicated transfer of energy, momentum and coherence from the electrons of the incident beam to the backscattered electrons [7,8].

Similar effects as in EBSD can be seen in Kikuchi patterns observed in transmission electron microscopy (TEM). It has been found that TEM Kikuchi patterns show a systematic dependence on sample thickness and electron acceleration voltage [9–13]. With decreasing voltage and increasing sample thickness, transmission Kikuchi bands change from the excess type (high intensity band) to the deficit type (dark bands) [10]. These observations give a hint about the crucial role that is played by the crystal thickness relevant for diffraction.

This is why an important question with respect to the details of pattern formation in EBSD is how the depth distribution of the incoherently backscattered electrons is influencing the contrast...
and sharpness of the observed Kikuchi patterns. First results in the direction of quantitative simulations of EBSD patterns were obtained recently by applying a Bloch wave approach and using the reciprocity principle for calculating the diffraction pattern of internal point sources of backscattered electrons [14,15]. In these investigations, thickness dependent effects were not addressed in detail. As we will see in this paper, the inclusion of the relevant thickness-dependent influences in the framework of the previous dynamical simulations consistently leads to Kikuchi band contrast reversal effects similar to those experimentally observed by Alam et al. [6].

In this paper, we present a combined experimental and theoretical study of incident beam effects on the Kikuchi band contrast in EBSD patterns from silicon. Specifically, we analyze the origin of the contrast reversal of Kikuchi bands that is observed in EBSD when changing the incidence angle of the primary beam [6]. We will first present the experimental data, then we discuss the inclusion of the depth distribution of the backscattered electrons in the theoretical framework, and we close with the comparison of experiment and simulation.

2. Experimental results

We measured EBSD patterns from a Si(111) sample at 20 kV using a LEO Gemini 1530VP scanning electron microscope and a HKL Technology Nordlys II EBSD system. The sample could be rotated around an axis perpendicular to a plane which includes the incidence direction and the direction to the pattern center (i.e. the plane of Fig. 1). Experimental patterns were taken for incidence angles from 52° to 72° with respect to the Si(111) sample surface normal. This changes the range of crystal directions which is visible on the phosphor screen. The pattern center direction is orientated at 93° with respect to the incident beam direction (see Fig. 1).

The experimental patterns are shown in Fig. 2. At 52° incidence angle, we can see the sample edge at the lower border of the picture. For this incidence condition, the Kikuchi bands show a characteristic change in contrast from the upper part of the pattern, where the bands show maximum intensity in their middle (appearing as excess bands), to the lower parts, where they show a minimum of intensity in the middle (appearing as dark deficit bands). The upper parts of the EBSD pattern correspond to electrons leaving the sample nearer to the surface normal, while the lower parts show electrons that leave the sample at grazing emission angles near the sample surface plane. With more shallow incidence angles (larger incidence angles with respect to the surface normal), the part of the patterns that shows the contrast reversal is decreasing, and at 68° and 72°, the Kikuchi bands appear completely as excess bands.

The observed changes in the contrast reversal effect must be related to the changing incidence geometry of the primary beam. We can see this by observing the [111] zone axis on the middle vertical of the patterns shown in Fig. 2 (explicitly marked in the pattern for 68° incidence). The position of this zone axis corresponds to a fixed electron exit angle with respect to the sample surface normal. We see that the contrast reverses with changing incidence angle for this constant exit direction: the zone axis is dark at 52° incidence (111) is in the center of the pattern), whereas it has high intensity at 72° incidence (111) is at the lower middle of the pattern). For a constant incidence angle, the strength of the contrast reversal is clearly governed by the exit direction, as we see in Fig. 2, where the contrast reversal is clearly stronger in the lower parts of the patterns (especially visible in the upper row of pictures).

Fig. 1. Experimental geometry used in the present study. The angles of incidence were changed by sample rotation from (a) 52° to (b) 72°, with the intermediate values shown in Fig. 2. Note that for different incidence angles, different crystallographic directions are mapped to fixed observation directions A and B on the phosphor screen, due to the fixed relative geometry of the microscope column and screen. The sample edge, corresponding to electrons with grazing emission angles, can be seen near B for incidence angles near 52°.

The observed changes in the patterns from Si are consistent with previous results of Alam et al. [6], who also observed inverted Kikuchi bands near grazing emission angles when the incident beam impinges more steeply onto the sample surface (Figure 9 in [6], NaCl at 50° incidence with respect to the surface normal). In the following section we will present a theoretical model which can be used to simulate this contrast reversal effect.

3. Theoretical model

We assume that the formation process of an EBSD pattern can be divided into three main stages: (1) elastic and inelastic scattering of the incident electrons, including channeling of the incident beam, (2) incoherent reversal of direction by a localized elastic scattering event (including recoil of the atom involved) and (3) diffraction of the point source electron wave by the surrounding crystal, including inelastic processes in the outgoing path. A full and general simulation of this problem has not been accomplished yet, but it has been demonstrated that under some simplifying assumptions one can simulate EBSD patterns on the basis of a model which employs a Bloch wave approach [14,15].

In short, these simplifying assumptions amount to assuming that the EBSD pattern is formed by electrons of different energies, emitted from the positions of the backscattering atoms. The depth distribution of the localized sources of backscattered electrons at each energy is assumed to be known as a result of a model for stage (1) of the process. The diffraction pattern at each energy is then calculated by using the reciprocity principle for a time-reversed plane wave incident on the sample from a direction on the observation screen. The total diffraction pattern is given by the integral of patterns at the respective energies multiplied by their spectral weight in the electron energy loss spectrum. In this approximation, the
The Bloch wave calculation then finds the coefficients $c_j$ by solving a matrix eigenvalue problem derived from the Schrödinger equation by limiting the wave-function expansion to a number of Fourier coefficients labeled by the respective reciprocal lattice vectors $g$, each of which couples the incident beam to a diffracted beam. The eigenvalues $\lambda^{(j)}$ appear when writing the Bloch wave vector $k^{(j)}$ as the sum of the inelastic processes in stage (3) are assumed to remove electrons from the elastic Kikuchi pattern of the primary point source that was formed at the backscattering atom in stage (2). Any additional diffraction effects of these inelastically scattered electrons that go beyond a self-consistent contribution to the incoherent depth distribution of stage (1) are neglected by the present model. For the processes to be discussed in this paper, and the previous investigations [14,15], this approximation turned out to lead to convincing agreement of the dynamical simulations with a number of experimental Kikuchi patterns from different materials.

In order to analyze our experimental observations shown in Fig. 2, we extended the previous framework for dynamical simulation of EBSD patterns [14,15] to include (a) the effect of a varying depth distribution of the backscattered electrons (e.g. as a simulation of EBSD patterns [14,15] to include (a) the effect of a stage (1) are neglected by the present model. For the processes to be discussed in this paper, and the previous investigations [14,15], this approximation turned out to lead to convincing agreement of the dynamical simulations with a number of experimental Kikuchi patterns from different materials.

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Fig. 2. Experimental EBSD patterns from single crystal Si(1 1 1), 20 kV. Indicated are the angles of incidence of the primary electron beam with respect to the surface normal of the sample. The raw experimental patterns were divided by a smooth background. The contrast and brightness were adjusted for all patterns in the same way to increase visibility of the discussed effect: (a) 52°; (b) 56°; (c) 60°; (d) 64°; (e) 68° and (f) 72°.

Fig. 3. Definition of the effective thickness $t = t_s / \cos \theta_{OUT}$ for backscattered electrons observed at an angle $\theta_{OUT}$ with respect to the surface normal and originating at depth $t_s$ below the sample surface.

The Bloch wave approach is used to describe the diffraction from internal sources by applying the reciprocity principle for a time-reversed plane wave incident from a direction on the observation screen [14,15]. The wave function in the crystal is expanded as [16,17]

$$\Psi(r) = \sum_j c_j \exp[2\pi ik^{(j)} \cdot r] \sum_g c_g^{(j)} \exp[2\pi i g \cdot r]$$

(1)

The Bloch wave calculation then finds the coefficients $c_j$, $c_g^{(j)}$, and the vectors $k^{(j)}$ by solving a matrix eigenvalue problem derived from the Schrödinger equation by limiting the wave-function expansion to a number of Fourier coefficients labeled by the respective reciprocal lattice vectors $g$, each of which couples the incident beam to a diffracted beam. The eigenvalues $\lambda^{(j)}$ appear when writing the Bloch wave vector $k^{(j)}$ as the sum of the incident beam wave vector $K$ in the crystal and a surface normal component as $k^{(j)} = K + \lambda^{(j)} n$, with a complex $\lambda^{(j)}$ in the general case. The Bloch wave simulation gives the unknown parameters defining the wave function in Eq. (1) for a given incident beam direction $K$ and electron acceleration voltage. We obtain the probability density $P(r) = \Psi(r) \Psi^*(r)$ at every point inside the crystal from (1) as

$$P(r) = \sum_{gh} \sum_j c_j^* c_g^{(j)} \exp[2\pi i (\lambda^{(j)} - \lambda^{(h)}) \cdot r] \exp[2\pi i (g \cdot h) \cdot r]$$

(2)

For a backscattering process starting at depth $t_s$ below the surface, the effective depth $t$ takes into account the tilt angle $\theta_{OUT}$ of the outgoing direction with respect to the surface normal $t(\theta_{OUT}) = t_s / \cos \theta_{OUT}$ (see Fig. 3). This means that electrons which leave the crystal at larger angles experience an effectively larger depth in the dynamical diffraction effects [17, Section 3.3].

The probability density $P(r)$ describes the relative intensity in the observed direction $K$ on the observation screen, starting from a specific position $r$ in the crystal (or by reciprocity, the probability of diffraction from an incident direction $K$ to a
position \( \mathbf{r} \) in the crystal). This means that \( P(\mathbf{r}) \) handles the modulation of backscattering from point sources of unit strength due to interference of scattered electron waves in the crystal structure. For a comparison with experiment, we thus have to integrate over all points in the crystal and weight them by the depth distribution \( \rho(t_z, \theta_{IN}) \) of backscattered electrons, which is created by the primary incident beam:

\[
I_{ERSD} = \int dr P(\mathbf{r}) \cdot \rho(t_z(\mathbf{r}), \theta_{IN})
\]

We assume that the depth distribution of backscattered electrons \( \rho(t_z, \theta_{IN}) \) is produced by the incident beam depending on the incidence angle \( \theta_{IN} \) and that \( t_z \) is measured along the sample surface normal.

4. Results and discussion

We carried out dynamical electron diffraction simulations for Si using the above improved model. Instead of trying to include the depth distribution of the backscattered electrons in all details, we concentrate on bringing out the main effect and assume two simplified cases: in one case, electrons come only from a shallow surface region, while in the other case, the electrons are assumed to originate from a buried slice of finite thickness below the surface. This is illustrated in Fig. 1 by the solid black bars at different depths below the sample surface.

Specifically, we assumed two simplified model cases: (a) the backscattered electrons are created with equal intensity from depths of \( 0 \ldots 40 \) nm for the incidence angle at \( 72^\circ \): \( \rho(0 \ldots 40 \text{ nm}) = 1 \), or (b) they are created in a layer \( 45 \ldots 60 \text{ nm} \) below the surface: \( \rho(45 \ldots 60 \text{ nm}) = 1 \) for the incidence angle of \( 52^\circ \). These numbers have been chosen for good agreement of simulation and experiment (see below) while keeping them compatible with an estimation of the electron elastic mean free path \( \lambda_e \approx 17 \text{ nm} \) at 20 kV in Si [18] and the assumption that the direction reversal of quasi-elastically backscattered electrons takes place by a single large-angle elastic scattering event within path lengths of a few multiples of \( \lambda_e \) [19]. We expect that more precise depth distributions can be obtained in the future by incorporating Monte-Carlo simulations [20] into the dynamical diffraction framework. In such a combined and quantitative treatment, it would be interesting to investigate the values of the elastic mean free paths that are usually given for amorphous materials in the literature. This would be important for a quantification of the information depth in electron scattering measurements from crystalline surfaces. However, in order to show in the simplest manner under what kind of fundamental assumptions the observed contrast reversal effects appear, we use the above defined model distributions for the purposes of the current paper.

In the dynamical simulation, the Si lattice constant assumed was \( d_B = 0.543 \text{ nm} \), we included 143 beams with maximum lattice spacing \( d_B > 0.095 \text{ nm} \), and the Debye–Wallner factor was \( B_B = 0.003 \text{ nm}^2 \). The Fourier coefficients of the real and imaginary (absorptive) parts of the crystal potential were calculated using the FSCATT subroutine of Weickenmeier and Kohl [21].

The results of the simulations are compared to the experimental patterns in Fig. 4. We see that the characteristic effect of Kikuchi band contrast reversal is faithfully reproduced by the dynamical model: for the steeper incidence angle (\( 52^\circ \), lower part), the Kikuchi bands reverse contrast for those electrons that exit at shallower angles. Also, the intensity reversal in the \( [1 1 1] \) zone axis is correctly reproduced. We see that our model successfully captures the physics behind the contrast reversal under a change of the incidence angle and the resulting change in the depth distribution of the backscattered electrons.

The simulations shown above reveal the physical mechanism behind the contrast reversal effect under simple and reproducible assumptions. We did not attempt a quantitative reproduction of the observed patterns in all their details. In particular, we note that the fine structure of the patterns is not fully reproduced due to the reduced number of reflections that was taken into account (for the level of detail that can be reproduced by the dynamical theory see e.g. [14,15,24]). Also, we did not include the effect of excess-deficiency lines. This effect can be seen in the experimental patterns of Figs. 2 and 4 as the strong asymmetry for the horizontal Kikuchi bands, showing high intensity at the upper edges compared to low intensity at the lower edges. A phenomenological model for inclusion of the excess-deficiency effects in dynamical simulations has been introduced previously [15].

The fundamental mechanism behind the thickness dependent Kikuchi band contrast is well known from work in transmission electron microscopy as the effect of anomalous absorption [22]. As we can see in Fig. 1, there is a distinctive difference between the two geometries: at steeper incidence (\( 52^\circ \)), the backscattered electrons are created deeper inside the sample on average [23]. A simple geometrical argument shows that at \( 72^\circ \), the incident beam has to penetrate about two times more material to end up at the same depth than at \( 52^\circ \), \( 1/\cos 72^\circ = 3.2, 1/\cos 52^\circ = 1.6 \). Thus it will be less likely for the \( 72^\circ \) incident beam to excite backscattering processes with the same intensity in the same depth as compared to \( 52^\circ \). However, the incident beam penetration effect by itself is not sufficient to cause a contrast reversal because we still observe excess bands in the upper parts of the Kikuchi pattern at \( 52^\circ \) incidence. What is seen is the varying amplification of the depth effect in the outgoing path. Electrons at grazing emission must be transmitted through a much larger amount of material than the electrons emitted near the surface normal direction. Thus, for the electrons at grazing emission, the crystal looks effectively thicker. Accordingly, they experience increased inelastic scattering. A part of the inelastic processes is concentrated near the atomic cores (e.g. thermal diffuse scattering). As also the relative position of the nodes and antinodes of the excited Bloch waves with respect to the atomic positions is characteristically different in the crystal near a Kikuchi band for directions smaller or larger than the Bragg angle, the localized
inelastic processes affect both types of Bloch waves to a different degree. The outgoing electrons nearer to the middle of a Kikuchi band travel preferentially at the atomic positions [24], and this is why they also experience increased thermal diffuse scattering. This leads to the effect that for thicknesses in the order of a few times the inelastic mean free path, the electrons in the middle of a band will have less intensity than the electrons at the edges of a band [22].

We would like to emphasize that the anomalous absorption effect is in principle always present in the process of EBSD pattern formation. In a depth-resolved EBSD pattern, the electrons from larger depths would always show the dark deficit Kikuchi bands. Under standard conditions of EBSD measurements, however, the contribution of these electrons to the final pattern can usually be neglected. This is because the EBSD pattern is integrating over an extended depth range and it is difficult to select a specific depth for measurement. Under standard measurement conditions for largest signal-to-noise ratio, large incidence angles are required to exploit the forward peaked scattering cross sections, and thus the detected electrons are backscattered from a rather shallow surface region. In this way, the lower depths are emphasized by a large incidence angle. When using steeper incidence, however, the overwhelming contribution from the region nearer to the surface is reduced and the deeper regions are emphasized. As we have discussed above, this is a requirement for the observation of the dark Kikuchi bands in EBSD patterns.

5. Summary

In this paper we investigated the origin of the observed contrast reversal in Kikuchi bands when the incidence angle is changed in EBSD experiments. We find that the depth distribution of the backscattered electrons combined with localized inelastic scattering processes in the outgoing path plays a decisive role in the contrast reversal. Backscattered electrons originate from deeper within the sample when the primary beam is incident at steeper angles to the sample surface. As we showed by comparison to dynamical calculations, this effect has direct consequences for diffraction patterns of electrons backscattered from larger depths in the sample. These electrons see an effectively thicker crystal with a correspondingly higher probability of inelastic scattering. Localized inelastic processes cause a preferential absorption of electrons that move in the directions near to the middle of a Kikuchi band. This leads to the development of a central intensity minimum and thus a dark Kikuchi band for the electron emitted at grazing angles with respect to the surface plane.

The complexity of the Kikuchi band contrast reversal effect underlines that the dynamical electron diffraction theory is crucial to understand the intricacies of pattern formation in EBSD in full detail.

References