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Theory and application of electron channelling contrast imaging under controlled diffraction conditions

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Abstract

Electron channelling contrast imaging (ECCI) is a powerful technique for observing crystal defects, such as dislocations, stacking faults, twins and grain boundaries in the scanning electron microscope. Electron channelling contrast (ECC) is strongest when the primary electron beam excites so called two-beam diffraction conditions in the crystal. In the present approach this is achieved, by a combination of crystal orientation measurement using electron backscatter diffraction (EBSD) and simulation of electron channelling patterns. From the latter, the crystal is rotated such that two-beam diffraction conditions are achieved. This technique is called "ECCI under controlled diffraction conditions" or cECCI. Following an extensive literature review, this paper presents a simple, yet instructive and demonstrative treatment of the theory of ECC of lattice defects based on Bloch wave theory using a two-beam approach. This is followed by a discussion of technical issues associated with an ideal ECC set-up such as optimum detector position and microscope conditions. Subsequently, the appearance of different types of lattice defects under ECCI conditions; namely of dislocations, stacking faults, slip lines, and nanotwins, is discussed in detail. It is shown how different types of defects are distinguished and which type of crystallographic information can be extracted from such observations. Finally, the limits of the technique, particularly in terms of spatial resolution and depth of visibility are discussed and a comparison with the EBSD and transmission electron microscopy techniques with respect to imaging lattice defects is provided. In contrast to many investigations recently published in the literature, the current paper focuses on 'true' backscattering, i.e. on a signal that is recorded with a conventional backscatter detector positioned below the pole piece, and not on forward scattering, where the signal is recorded on a detector usually positioned below the EBSD detector. This has significant advantages in terms of spatial resolution and contrast, which are discussed in the text.

Keywords: Electron channelling; Electron backscatter diffraction; Bloch wave theory; Dislocation twinning

1. Introduction

Scanning electron microscopy (SEM) provides detailed information on surface topography via secondary electrons (SE), and surface chemistry via backscattered electrons (BSE), energy or wavelength dispersive X-ray spectroscopy and cathodoluminescence [1,2]. In addition, SEM can provide crystallographic information via electron backscatter diffraction (EBSD) (e.g. Ref. [3]). While it has been proved to be feasible in scanning electron microscopes by electron channelling contrast (ECC), the direct observation of crystal lattice defects is commonly thought to be the domain of transmission electron microscopy (TEM) performed on thin foils. With a new generation of SEM instruments that provide concurrent small beam convergence, high current density and small beam diameter, ECC enables the convenient, direct observation of dislocations, stacking faults (SF), nanotwins and elastic strain fields in bulk samples. One of the main aims of this overview is to exemplify electron channelling contrast imaging (ECCI) as an elegant,

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simple-to-conduct method for observation of lattice defects in bulk samples.

First, this text provides an overview of the historical development of the technique, before focusing on theoretical and practical aspects of the technique. In the theoretical treatment of ECCI presented in Section 3, the Bloch wave treatment of dynamical electron diffraction is employed to explain the contrast obtained from lattice defects such as SF and dislocations. The theoretical treatment is employed to elucidate optimum imaging conditions and to evaluate the limitations of imaging in Sections 4 and 5. Subsequently, in Section 6, the latest results of detailed dislocation and SF analysis using ECCI under controlled diffraction conditions are presented and analysed. Two types of steel samples were investigated: a steel with twinning-induced plasticity (TWIP) with face-cantered cubic (fcc) crystal structure, and an electrical steel with bcc crystal structure. The TWIP steel has a low SF energy and therefore tends to form planar slip features (slip bands), SF and twins. The deformed electrical steel shows screw dislocations. The paper concludes in Section 7 with an overview of the advantages and limitations of the ECCI technique compared with competing EBSD and TEM techniques.

2. Overview of the literature on channelling contrast of crystal defects

The most comprehensive overview of the principles and historical foundations of the channelling contrast technique remains that published by Joy, Newbury and Davidson in 1982 [4]. In comparison, the present authors review here only a selection of publications, also covering some of the developments in the 30 years following the work of Joy et al.

Coates [5] (with comments by Booker et al. [6]) was the first to observe Kikuchi-like reflection patterns superimposed on the topographical image obtained with BSE. These patterns, appearing on the surface of bulk single crystals at low magnifications, were similar in geometry to backscatter Kikuchi patterns (as observed first by Nishikawa and Kikuchi [7]) and obviously carried crystallographic information. At low magnifications, the angle between the incident beam and the surface normal varies appreciably over the field of view. For those positions where the incident beam satisfied the Bragg position for a particular set of crystal lattice planes, low backscatter intensity resulted. As a consequence, dark lines corresponding to Kikuchi lines were visible across the specimen surface. The low backscatter signal was interpreted in terms of channelling of the primary beam electrons along particular crystal directions. The resulting patterns were therefore termed "electron channelling patterns" (ECP). Alternatively, similar patterns could be produced by rocking the beam over a fixed point on the sample and recording the backscattered electron intensities independent of the primary beam incidence angles [8,9].These were termed either ECP or "selected area channelling patterns". Every direction in such a pattern corresponds to one well-defined beam-incidence direction on the crystal, and the centre of the pattern corresponds to the beam incidence direction that the primary beam takes during normal image-mode scanning.

The variance of backscattering intensity observed with respect to changes in crystal orientation led to the conclusion that near-surface defects, which change the crystal orientation locally, should also lead to a change in the BSE intensity, and hence should be detectable [10,11]. Intensity profiles of BSE, when mapped across defects such as screw dislocations and SF, were calculated and resulted in well-defined intensity profiles [12,13]. Based on these calculations, imaging of defects in BSE mode became feasible [14–16], but most of the images were taken without any control of the imaging conditions. This was changed by Morin et al. [17], who combined the recording of ECP using a rocking beam method with observation of the BSE images after an appropriate tilting of the specimen. The sample was tilted such that a dark Kikuchi line in the ECP runs through the centre of the pattern, which indicated channelling conditions for the normal scan across the sample at high magnification. These authors also the made first attempts to observe the contrast change in dislocations related to different diffraction conditions. Three comprehensive reviews on channelling contrast and on backscattering were presented at that time by Joy et al. [4], Niedrig [18] and Lloyd [19]. Using a forward-scatter arrangement, Czernuszka et al. [20] observed dislocations in silicon and Ni_3Ga, and Wilkinson et al. [21] imaged and simulated dislocations in silicon–germanium layers. In contrast, Ng et al. [22] used a backscatter setup to image individual dislocations at crack tips in NiAl. Zauter et al. [23] and Ahmed et al. [24] demonstrated the visibility of dense dislocation walls. The latter is easier than observation of individual dislocations because dislocation walls show a constant bright contrast independently of the orientation of the crystal with respect to the electron beam. This was explained by a model considering the effect of kinks in the dislocation lines on the electron backscattering behaviour developed by Dudarev et al. [25].

Further technological developments, in particular thermal field emission guns with high probe current in a small probe diameter, semiconductor BSE detectors with higher sensitivity, and more convenient stage configurations, allowed for better channelling contrast recording. Significant work was then done by Crimp et al. [26,27], who accomplished tests on the invisibility criterion of edge dislocations in Fe–50%Al, and on dislocation density measurements in 2% rolled Ti. More recently, intensive work was done on the characterization of threading dislocations in thin films, particularly in different carbides and nitrides, e.g. [28–33]. Note that most of these studies used forward scattering rather than backscattering, which has consequences that will be discussed in Section 4.3. Picard, Twigg and others [28,29] showed that the determination of the