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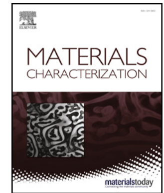

Space rocks and optimising scanning electron channelling contrast

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ABSTRACT

Forescatter electron imaging is a popular microscopy technique, especially for scanning electron microscopes equipped with an electron backscatter diffraction detector. In principal, this method enables qualitative imaging of microstructure but quantitative assessment can be limited due to limited information about the contrast afforded. In this work, we explore forescatter electron imaging and demonstrate that imaging can be optimised for topographic, phase, and subtle orientation contrast imaging through appropriate sample and detector positioning. We demonstrate the relationship between imaging modes using systematic variation in detector positioning and compare this with pseudo-forescatter electron images, obtained from image analysis of diffraction patterns, to explore and confirm image contrast modes. We demonstrate these contrast mechanisms on a map obtained from a sample of the Gibeon meteorite.

1. Introduction

Imaging in the scanning electron microscope is a popular method of gaining insight into the microstructure, phases, and sub-structure of crystalline materials. Electron backscatter diffraction (EBSD) can be an excellent method of quantified understanding of crystalline materials, as at each interrogation point a diffraction pattern can be captured for analysis, either online or offline, to reveal local crystal orientation, phase and even variations in elastic strain [1]. However, this method can be slow and often requires *a priori* knowledge of the microstructure to be examined, such as the phases or grain size for targeted quantitative assessment. This promotes the use of fast imaging modes, such as backscatter or forescatter (i.e. forward scattering) electron imaging (where ‘back’ and fore- aka ‘forward’ refer to the path of the scattered electrons used to generate image contrast, with respect to the incident beam). This technique has seen an increase in popularity recently, such as the generation of virtual scattering images from post processing of the intensity distributions collected on the scintillator-coupled-CCD EBSD detector [2] which has resulted in the PRIAS technology from EDAX-TSL. However, the virtual technique can be limited by the read-out and efficiency of scintillator-to-CCD read out and interpretation of these images requires knowledge of the electron scattering processes and image processing steps.

Use of semiconductor based, i.e. hardware, electron imaging technologies such as diodes have been incredibly useful in the SEM environment and many of these are supplied by microscope manufacturers. Furthermore, extra diodes are often introduced onto the front of the EBSD detector, motivated largely by the early work of Day and Quested [3] and realised also in work by Prior et al. [4]. In these geometries, silicon diodes are introduced to provide images from highly tilted samples and the signal from these diodes is optimised to provide rich microstructural images that highlight microstructural features.

The yield of electrons that reach a detector in an electron microscope is a function of several processes combined:

(1) The formation of a near parallel beam and how it is scanned across the sample (including dwell time, focus and probe current).
(2) Electron entry and escape – where the topography and inclination of the surface (both as the electrons enter, and as they leave) influences the yield of electrons.
(3) Electron channelling-in - where by the depth and scattering of the electron within the sample is controlled by the orientation (and phase) of the sample within the interaction volume [5].
(4) Electron scattering & channelling out - where the path can be perturbed by near-elastic electron interactions with the crystal lattice, which is the origin of the Kikuchi bands of raised intensity with the electron backscatter diffraction pattern [6].
(5) Electron scattering & effective density – where the scattering efficiency is proportional to the electron density of the material within the interaction volume, giving rise to atomic number (i.e. *Z*-contrast) and variable electron energies in the escaping electrons [5].
(6) The position, size, and form of the diode(s) with respect to the sample – which can bias the signal due to variable emission, detection efficiency, noise, and angle subtended by the diode(s) [7].

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(7) Voltage of the incoming beam, as this will affect the depth of penetration, efficiency of detection, and spatial resolution (due to electron optics); as well as channelling-in and channelling-out behaviour (due to diffraction effects related to the wavelength of the electron beam).

These processes affect each other in turn, and rarely can be considered entirely in isolation. It can be useful to separate them here (e.g. the separation of channelling in and out is slightly artificial) and this can aid in interpreting the information obtained.

In the present work, we will not address (1) strongly, as in general there is limited flexibility within most scanning electron microscopes.

For the majority of the analysis in this work, we have selected to tilt the sample strongly towards the detector (tilting up to 70°) and have tilted it significantly towards the EBSD detector. This enables us to best present the sample to diodes mounted near to the EBSD detector, as well as enabling direct comparison with the electron cloud which is incident on the EBSD detector. In general, tilting the sample introduces significant imaging distortions [8]. Furthermore, this can make physical analysis of the electron path more complex (as the perpendicular introduction of an electron probe into a semi-infinite lattice-structure half space is easier to compute), but according to Reimer [9] and in the simulations shown in Payton and Nolze [10] there is a strong increase in emission of electrons from highly tilted samples.

Within the literature there is significant discussion on the nature of contrast in back and forward scattering imaging, and the role of electron channelling-in and channelling-out. This has resulted in differences in opinion for the optimum position of the detector [7, 11–13] to optimise contrast. In the present work, we will focus our study to lower magnification images of sub-structure which highlight low angle grain boundaries, topography, and grain structure. We will not focus on optimised contrast for dislocation analysis.

The present work links to both the excellent single crystal analysis of Winkelmann et al. [7], who explore the role of channelling-in and channelling-out within single crystal semiconductors; as well as the identification of the relationship between channelling-in and channelling-out contrast due to the effect of crystal rotations of Kaboli and Gauvin [13]. Importantly for the present study, Winkelmann et al. [7] show that the electrons received with a virtual detector placed towards the top of the EBSD phosphor screen highlight terracing on a single crystal growth surface, whereas virtual detectors placed towards the bottom of the EBSD phosphor screen highlight local strain and orientation variations due to threading growth dislocations. The benefit of virtual detector analysis is also highlighted in the work of Nolze et al. [14] who explore a range of detection modalities using (largely) electron backscatter pattern (EBSP) based approaches, including a specific note that a significant amount of contrast within virtual FSD detectors is common between the raw EBSP analysis and analysis of only the background signal (and thereby also supporting an assertion that a significant amount of contrast within FSD images is from channelling in phenomena).

In this manuscript, we explore electron channelling contrast using a sample from the Gibeon meteorite. This meteorite fell in prehistoric times over an area of 275 km near the village of Gibeon within the Hardap Region of Namibia. The sampled area of this meteorite is an iron-nickel rich microstructure, with very large Widmanstatten structures due to the exceptionally long cooling periods. These structures are likely formed as the meteorite cools from homogeneous austenite phase to the austenite + ferrite phase field with a likely cooling rate of a few hundred degrees per million years [15]. This cooling rate results in the generation of multiple low angle grain boundaries within the microstructure, and an orientation relationship between the body centred cubic (ferrite, aka kamacite) and face centred cubic (austenite, aka taenite) [16]. We have selected this sample from a technical perspective as it has low angle grain boundaries, interrelating orientation relationships, and variable chemistry; and it is both aesthetically pleasing to work with and exciting to probe near equilibrium microstructures formed in asteroidal bodies.

2. Materials and Methods

A sample of the Gibeon meteorite (purchased from eBay and kindly supplied by Peter Eschbach from Oregon State University) was metallographically polished to a high quality finish, with an ultimate step using colloidal silica. The sample was cleaned using an in-chamber plasma cleaner to reduce carbon contamination from repeat imaging. Microscopy was performed at 20 keV on a Zeiss Merlin FE-SEM using a probe current of ~7 nA. Forescatter electron imaging was captured using the ARGUS imaging system, mounted on a Bruker *e*-Flash FS EBSD detector and EDS data was acquired using a Bruker XFlash 6|60 detector.

Repeat forescatter electron imaging was performed for three experiments, exploring: (1) optimum detector angle, using detector tilt; (2) optimum detector distance (i.e. angle subtended for the exit electron beams on the sample); (3) variation in contrast to highlight channelling-in contrast.

The forescatter electron imaging has been compared with virtual detectors formed from selected area analysis of captured EBSD patterns. These patterns were captured with an *e*-Flash FS detector in 2 × 2 binning (320 × 240 pixels) and with zero gain camera settings and stored to disk. The detector was placed with a pattern centre of [0.54, 0.50, 0.64] and there was a detector tilt of 5.27° (for a description of the conventions used here, please see [17]).

Online analysis of phase and crystal orientation was performed using ESPRIT v2.1, with the austenite and ferrite phases selected to index the taenite and kamacite phases respectively.

EDS analysis was performed with cluster analyse based upon the EDS spectra captured. This was performed with a histogram analysis algorithm in ESPRIT 2.2. Software parameters of a sensitivity of 81 and an area setting of 0.35% were used with this clustering algorithm to empirically optimise segmentation and clustering of the minor phase (taenite).

To reduce map distortion, all maps were spatially registered against a normal incidence backscatter image (captured with a detector mounted on the pole piece) of the same microstructure taken with perpendicular incidence of the incoming electron beam. All qualitative image maps are presented in the corrected frame (correction has been performed using a bicubic interpolation of the relevant map in colour space). EBSD orientation data was registered and corrected using nearest neighbour interpolation. All maps were cropped to the same field of view.

Image processing of the EBSD patterns was performed for the virtual FSD experiments. The background was fit with a Gaussian function using a linear minimisation function with Matlab, fitting Eq. (1) for the intensity distribution within each diffraction pattern:

$$I = I_b \exp\left\{\frac{-(x-x_c)^2}{2x_w^2} + \frac{-(y-y_c)^2}{2y_w^2}\right\} \tag{1}$$

where the fitting constants include: I_b = scaling of the background intensity; (x_c, y_c) is the centre of the Gaussian; and x_w and y_w are the Gaussian widths in X and Y respectively.

Once the intensity of the background was fitted, a background corrected pattern was obtained by dividing the raw image by the fitted background function.

For computational speed, fitting of this Gaussian background was computed for each pattern after software binning to 160 × 120 pixels (and therefore the fitting functions were calculated for this image array size).

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