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High-resolution scanning precession electron diffraction: Alignment and spatial resolution

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ABSTRACT

Methods are presented for aligning the pivot point of a precessing electron probe in the scanning transmission electron microscope (STEM) and for assessing the spatial resolution in scanning precession electron diffraction (SPED) experiments. The alignment procedure is performed entirely in diffraction mode, minimising probe wander within the bright-field (BF) convergent beam electron diffraction (CBED) disk and is used to obtain high spatial resolution SPED maps. Through analysis of the power spectra of virtual bright-field images extracted from the SPED data, the precession-induced blur was measured as a function of precession angle. At low precession angles, SPED spatial resolution was limited by electronic noise in the scan coils; whereas at high precession angles SPED spatial resolution was limited by tilt-induced two-fold astigmatism caused by the positive spherical aberration of the probe-forming lens.

1. Introduction

The vast majority of technologically important materials, across myriad applications from electronics to structural engineering, are crystalline, or part crystalline, in nature. Many of the underlying physical, chemical and mechanical properties of these materials are dictated by the presence of crystal defects (e.g. grain boundaries, dislocations and strain) either by design or as a consequence of natural disorder. In either case, it is critically important to determine the crystallographic nature of the defects, often at the nanoscale, in order to understand the materials properties. As such, a technique is required to map changes in crystallography, with high spatial resolution and accuracy, which is adaptable to different materials and devices. Electron back-scatter diffraction (EBSD), performed in the scanning electron microscope, is extremely effective over length scales ranging from hundreds of micrometres down to several tens of nanometres [1], but the gap between ten nanometres and the sub-nanometre scale remains. Scanning electron diffraction (SED) in the transmission electron microscope (TEM) is emerging as a strong contender to fill this gap. The technique involves rastering a nanometre-sized electron probe over an area of interest and recording the transmitted diffraction pattern at each probe position. The rise in popularity of SED has been driven by recent developments in scanning transmission electron microscopy (STEM) including: high brightness electron sources [2], flexible probe-forming electron optics [3], fast, high dynamic range pixelated detectors [4–6]; as well as the availability of computational power to process the large four-dimensional (4D) data-sets [7] obtained.

In many applications of SED, the geometry of each diffraction pattern is measured, almost always with an automated procedure. Such analysis can be improved by combining SED with precession electron diffraction (PED). In the PED method, known originally as the double-conical beam-rocking method [8], the incident electron probe is tilted away from the optic axis by the precession angle, ϕ, which is typically 0.5–3°, and the tilt azimuth is rotated around the optic axis (Fig. 1). For a crystal with a zone axis parallel to the optic axis, the tilted beam expands the zero-order Laue zone (ZOLZ) from a point to a circle (diameter equal to 2ϕ) and rotating the azimuth sweeps the ZOLZ circle around the optic axis, exciting many reflections temporarily as they pass through the Bragg condition (twice) for each complete rotation of the azimuth. By de-rocking the diffracted beams below the sample, the circular movement of the reflections, observed in the diffraction pattern, is arrested. The net effect is equivalent to precessing the sample around a stationary electron beam [8]. There are two significant advantages of precession: the number of reflections increases as the Ewald sphere rocks through many more reciprocal lattice points; and the thickness-dependence of the ensemble of reflections appears to be slower, i.e. the reflections are 'kinematic-like' [9–11]. Together, this has made zone-axis PED patterns particularly amenable for structure solution problems [12–14].

Scanning precession electron diffraction (SPED) involves rastering a precessing probe over an area of interest and recording a PED pattern

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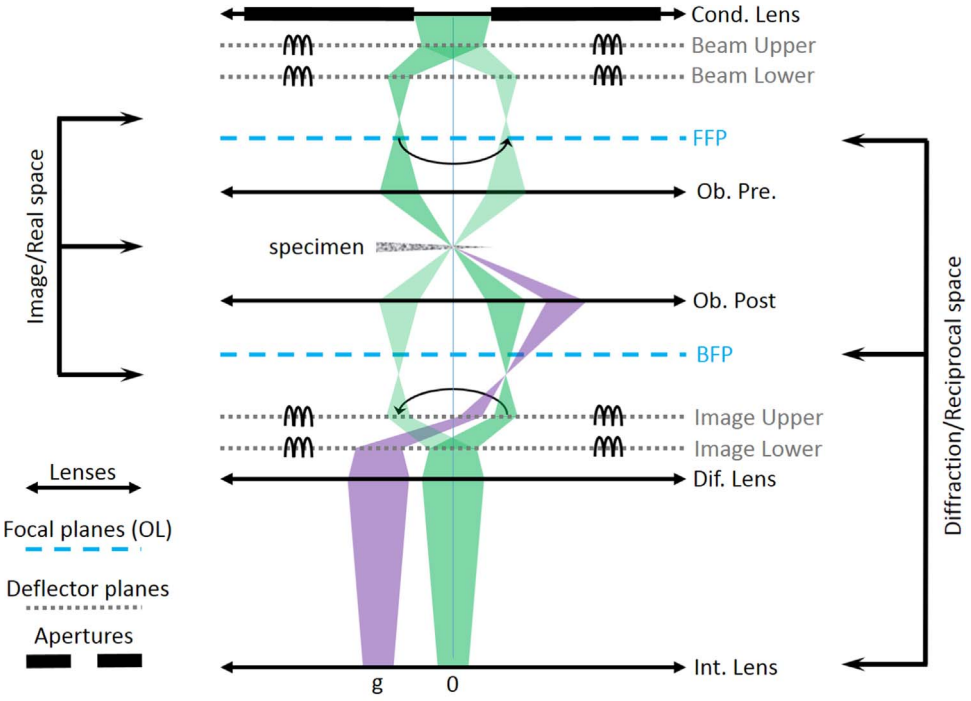

Figure 1

Fig. 1. Schematic of the focussed precessing probe geometry for SPED. Two azimuths (dark green and light green) are depicted for the direct beam, 0, with one diffracted beam, g, (purple) illustrated that shows the diffraction condition is only met at one particular azimuth. The Beam Upper and Lower Deflectors (depicted as inductors) shift and tilt the beam above the sample, the Image Upper and Lower Deflectors de-rock the diffraction pattern below it. Principal imaging and conjugate diffraction planes are indicated, as well as the principal focal planes of the objective lens (OL). Lenses labelled are: condenser (Cond.), diffraction (Dif.), intermediate (Int.) and objective pre (Ob. Pre.) and post (Ob. Post) specimen lenses. (Note: Colour available in the online version of this article.).

at each probe position. This has been implemented on a number of microscopes and has proved advantageous for orientation imaging [15–17], phase identification [18], strain mapping [19–22], and three-dimensional interphase crystallography [23]. Perhaps most striking is the improvement in precision for strain mapping, which is of key importance in the semiconductor industry where strain is introduced to increase carrier mobility [24]. The improvement in strain measurement in SPED is attributable to: i) more reflections being recorded in each PED pattern; ii) the presence of higher order reflections increasing the sensitivity to small strains; iii) the position of each reflection being more easily located, because each PED reflection is a uniformly filled convergent beam electron diffraction (CBED) disk [25]. Finally, it is noted that, with the addition of an EDX spectrometer, the fidelity of composition measurement is improved because unwanted channelling conditions areintegrated by the beam rocking [26].

For precession-enabled techniques (PED and SPED) the alignment procedure must accommodate the need to bring the precession pivot point into coincidence with the sample. If the STEM is fitted with an aberration corrector in the imaging (post-specimen) lens [19–22], then the image of the probe on the viewing screen is a reasonably accurate representation of the probe at the sample. Minimisation of probe wander on the viewing screen, with the sample in focus, is then a sufficient condition for achieving the correct pivot point for all but the highest precession angles. For non-image-corrected instruments, spherical aberration in the imaging lens and small misalignments between the pre-field and post-field objective lens pole pieces leads to significant misrepresentation of the probe position and shape on the viewing screen [27]. Aligning the pivot point in imaging mode can be successful if the probe movement relative to the sample is minimized [28]. However, it is emerging that the shadow image in a bright-field (BF) CBED disk, rather than the image of the probe, is a more appropriate representation for aligning the pivot point accurately [28]. This paper shows that the pivot point can be aligned entirely in diffraction mode, by using the shadow image in the BF-CBED disk. An explicit alignment procedure is provided and applied to obtain high spatial resolution SPED data sets. Physical limitations imposed by electronic noise in the scan coils and aberrations in the probe forming lens are also discussed.

2. Materials

All data presented here were acquired using an FEI/Philips CM300F TEM operated at 300 kV with a Schottky thermionic source and working in microprobe mode. Both scanning and precession were enabled through a NanoMEGAS Digistar system hardwired into the microscope scan control boards. The system was controlled through the NanoMEGAS ASTAR software package using a Stingray fast capture CCD camera to capture the diffraction patterns as seen on the small viewing screen of the microscope.

The proposed alignment procedure (Fig. 3) was demonstrated with a Ted Pella test sample (product number 673) comprising a carbon replica of a crossed diffraction grating (500 nm pitch), shadow-coated with gold-palladium and decorated with 262 nm diameter latex spheres. The probe illumination semi-angle (α) and the precession angle (ϕ) were measured to be 3.35 mrad and 16.6 mrad respectively. The camera length was 850 mm.

The spatial resolution in SPED was assessed (Fig. 4) using an Agar Scientific combined test specimen (Product number S142), comprising a holey carbon film (thickness, t = 20 nm) decorated with a uniform distribution of gold particles approximately 10 nm in diameter. SPED data comprising diffraction patterns with 144×144 pixels and 8 bits per pixel were acquired with a spatial sampling of 1.9 nm/pixel and 256×256 data points (486 nm square). Precession angles (ϕ) between 1.4 mrad and 41 mrad (calibrated in-situ using polycrystalline gold ring patterns) were used with alignment performed as described in this work and at a precession frequency of 100 Hz. All data were acquired with: an illumination semi-angle, α = 0.91 mrad; a probe current of 0.5