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Transmission Kikuchi diffraction versus electron back-scattering diffraction: A case study on an electron transparent cross-section of TWIP steel

Azdiar A. Gazder[a,*], Khalil I. Elkhodary[b], Mitchell J.B. Nancarrow[a], Ahmed A. Saleh[c]

[a] Electron Microscopy Centre, University of Wollongong, NSW 2500, Australia
[b] Department of Mechanical Engineering, The American University in Cairo, New Cairo 11835, Egypt
[c] School of Mechanical, Materials and Mechatronic Engineering, University of Wollongong, NSW 2522, Australia

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ABSTRACT

The present case study compares transmission Kikuchi diffraction (TKD) with electron back-scattering diffraction (EBSD) on the same area of an electron transparent cross-section of a twinning induced plasticity steel. While TKD expectedly provides better clarity of internal defect substructures in the band contrast map, EBSD returns orientation data that approaches the quality of the TKD map. This was rationalised by Monte Carlo simulations of the electron energy spreads, which showed that due to the geometry-based compromises associated with adapting a conventional EBSD detector (which is off-axis with respect to the incident electron beam) to TKD, a broadening in the electron energy distribution of the forward-scattered electrons collected on the detector phosphor screen, is unavoidable. In this circumstance, the values of the full-widths at half-maximum of the energy distributions for TKD and EBSD are of the same order. It follows that EBSD on electron transparent cross-sections may be a viable alternative to TKD when: (i) conventional EBSD detectors are adapted to TKD and, (ii) sample microstructures comprise features whose sizes do not mandate the application of TKD.

1. Introduction

Conventional electron back-scattering diffraction (EBSD) undertaken in a scanning electron microscope (SEM) returns orientation information at a spatial resolution limited to ~50 nm due to the large interaction volume generated by the electron beam within infinitely thick (bulk) samples. Additionally, maximising the collection efficiency of back-scattered electrons on to the detector phosphor screen requires sample tilting to 70°. This results in the approximately circular electron beam taking on an elliptical shape with an aspect ratio of ~2.92 when projected on to the sample surface (Schwartz et al., 2000); with the projected beam spread along its major axis reducing the spatial resolution of the map in the vertical direction. It follows that the characterisation of nanometer-sized structures remains outside the capability of EBSD (Schwartz et al., 2000).

Alternatively, the transmission Kikuchi diffraction (TKD) technique (Keller and Geiss, 2012) adapts EBSD hardware/software to return quantitative orientation information at spatial and angular resolutions that approach those of scanning-transmission electron microscopy (Trimby, 2012; Trimby et al., 2014). The order of magnitude improvement in resolution is realised by passing the beam through thin, electron transparent cross-sections produced as electro-polished foils or focused ion beam lamellae. This largely avoids the lateral spread of the electron beam caused by elastically scattered electrons. In TKD, the scatter profile of the interaction volume is a narrow cone-shaped region running through the sample thickness. At/near the sample bottom plane, the electrons undergo diffraction and are forward-scattered over an approximately conical angular range; following which the Kikuchi patterns are collected on the detector phosphor screen (Keller and Geiss, 2012).

Based on the above, TKD should ideally be undertaken with the sample perpendicular to the incident beam in order to maintain the circularity of the electron beam and minimise the interaction length/volume and beam spread before the electrons exit the sample bottom plane. Since most forward-scattered electrons tend to be concentrated along the direction of the incident beam (van Bremen et al., 2016), on-axis detectors provide an ideal solution by enabling the highest line-of-sight collection efficiency while simultaneously allowing for reduced probe currents and beam diameters (Fundenberger et al., 2016).

While on-axis setups involve either a major detector redesign or a relocation of the phosphor screen, the modifications are proprietary such that the latter solution recently developed by the Bruker Corporation (Fundenberger et al., 2016) is not available for EBSD systems from other manufacturers.

* Corresponding author.
E-mail address: azdiar@uow.edu.au (A.A. Gazder).

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Due to the above circumstances, in most cases, TKD continues to be undertaken using conventional EBSD detectors (which are off-axis with respect to the incident electron beam) that are optimised for orientation mapping in scanning mode. Consequently, the following paragraphs briefly summarise the compromises imposed on TKD by this geometry.

Firstly, a general TKD rule of thumb is to place the sample at relatively short working distances (~4–8 mm); a position that roughly coincides with the top of the detector phosphor screen. The major drawback of placing the sample in this position is that it raises the pattern centre to the top of the region of interest. This in turn leads to Kikuchi bands being wider than normal at the bottom of the pattern, possessing asymmetric intensities across band widths and/or suffering gnomonic distortion (Trimby and Klement, 2015). Since most conventional indexing algorithms cannot reliably solve such distorted Kikuchi bands, the area undergoing Hough transformation needs to be restricted to the undistorted region at the centre of the pattern or indexing algorithms need to be modified to account for the distortions (Trimby and Klement, 2015).

Secondly, when the detector is off-axis with respect to the incident beam (Suzuki, 2013), it results in the restricted collection of only those electrons that are forward-scattered at high angles (Fundenberger et al., 2016).

In practice, (a) minimising distortions in the Kikuchi bands during TKD by maintaining the pattern centre in a position similar to EBSD, while concurrently (b) mitigating low electron intensity, poor line-of-sight collection efficiency and improving EBSP indexing rates, can be achieved by: (i) increasing the working distance and, (ii) tilting either the detector or the sample towards each other. In the case of (ii), detector tilting cannot be readily accomplished without hardware modifications and/or compromising conventional scanning geometry whereas sample tilting is relatively easier to undertake. The exact working distance and sample tilt angle (ranging from −10° to −40° (Brodusch et al., 2013)) depends on the physical configuration of the microscope-detector combination (Suzuki, 2013). It should be noted that while sample tilting is a sub-optimal solution (similar to EBSD, the increased aspect ratio of the elliptical projected beam along its major axis reduces the spatial resolution TKD map in the vertical direction) compared to on-axis detectors, it remains the more feasible alternative for the reasons stated above.

Lastly, TKD tends to be highly sensitive to the thickness of electron transparent cross-sections (Rice et al., 2014) which can be produced as foils using the widely available electro-polishing technique or as lamellae via focused ion beam milling. In the case of foils, there is a greater degree of difficulty in controlling the final thickness of the electron transparent region and the associated wedge profile around the hole; which in turn, leads to higher sample attrition rates during TKD. Although sample cross-section thicknesses can be better controlled when ion beam milling lamellae, the mapped area remains limited in comparison to foils. While this poses no problems for nanometer-sized microstructures, it is not ideally suited for microstructures with micron-sized or mixed micron and nano -sized features.

In summary, although TKD is the only SEM-based orientation imaging technique for nanometer-sized structures (whose characterisation requires minimised beam scattering volumes, energy and angular distributions (Keller and Geiss, 2012)), there are practical compromises imposed on it when conventional EBSD detector setups are adapted to TKD.

Moreover, in the particular cases where the sample: (i) electron transparent cross-section is too thick for TKD, (ii) is beam sensitive such that the higher probe currents required for TKD are not an option or, (iii) microstructure comprises features whose sizes do not mandate the use of TKD, we suggest employing a simpler alternative.

In the present case study, we show that undertaking EBSD on electron transparent cross-sections (Keller and Geiss, 2012) returns orientation data that approaches the quality of TKD when the latter is undertaken by adapting EBSD detectors. We demonstrate this by comparing the distributions of band contrast, mean angular deviation, grain orientation spread and geometrically necessary dislocations from TKD and EBSD maps acquired from the same area of an electron transparent electro-polished foil.

In order to highlight the efficacy of the proposed approach, a deformed twinning-induced plasticity (TWIP) steel was deliberately chosen, as it comprises micron-sized grains with sub-micron sized deformation twins. In this regard, previous studies have pointed out that the thickness of individual deformation twins is of the order of tens of nanometres such that they cannot be crystallographically detected via EBSD on bulk samples (which can only index twins once they stacked into relatively thicker bundles) (Saleh et al., 2013).

Although Monte Carlo (MC) simulations are unable to estimate the small volumes from which Kikuchi bands originate (Winkelmann, 2010; Zaefferer, 2007), this case study nonetheless uses them in a limited capacity to provide a comparison of the energy distributions of the forward and back -scattered electrons during TKD and EBSD, respectively.

2. Experimental and analytical procedures

An Fe-24Mn-3Al-2Si-1Ni-0.06C (wt.%) TWIP steel was slab cast, hot rolled to 52% followed by 42% cold rolling reduction. A flat dog-bone tensile sample with 25 mm gage length, 5 mm width and 1 mm thickness was wire-cut from the cold rolled strip with the gage length and width parallel to the rolling (RD) and transverse (TD) directions, respectively. In order to obtain a fully recrystallised microstructure, the tensile sample was annealed at 850 °C for 540 s (240 s of heating to stable temperature and 300 s of soaking) followed by water quenching. Further processing details may be found in (Saleh et al., 2011, 2013; Santos et al., 2011). Uniaxial tensile testing was conducted on an in-house modified Kammrath & Weiss GmbH tensile stage operating in speed control mode at 5 μm s^{-1} up to a true strain of 48%; with the latter corresponding to an ultimate tensile strength of 1080 MPa.

Following this, the tensile sample was mechanically ground to 0.3 mm thickness using 1200 grit silicon carbide paper. Ø3 mm discs were punched out from the gauge length after ensuring that each disc contained a short chord parallel to the tensile axis, in order to identify the macroscopic sample coordinates in the SEM. The discs were manually ground to ~70 μm thickness and twin-jet electro-polished to produce electron transparent foils using a solution of 90% methanol and 10% perchloric acid in a Struers Tenupol-5 operating at 30 V (~150 mA) and −30 °C.

TKD and EBSD were undertaken on the same electron transparent area in a JEOL JSM-7001F field emission gun (FEG)-SEM equipped with a Nordlys-II(S) detector interfacing with the Oxford Instruments (OI) AZtec software suite.

In the case of TKD, the foil was placed in a JEOL Be single-tilt holder screwed to an adaptor that fits onto the FEG-SEM sample holder. For EBSD, the foil was mechanically clamped on either side using an in-house developed holder such that the bottom plane of the foil is clear of obstruction.

TKD was performed at 30 kV accelerating voltage, ~10 nA probe current, 12 mm working distance (WD) and 40° stage and −50° sample tilts. On the other hand, EBSD was conducted at 15 kV accelerating voltage, ~5.1 nA probe current, 12 mm WD and 70° sample tilt. Based on the manufacturer's specifications, for aperture size number 4 (size = 30 μm diameter), the probe diameter is d_P = 7.105 nm at 30 kV, 10 nA, WD = 12 mm during TKD and d_P = 7.326 nm at 15 kV, 5 nA, WD = 12 mm during EBSD.

During TKD, a higher accelerating voltage enables electrons to traverse the transparent cross-section while a higher probe current improves the signal-to-noise ratio. For our microscope configuration, the above WD and stage/sample tilts keep the pattern centre in the same position during TKD and EBSD. Software tilt correction was applied for both techniques.