Ultramicroscopy 135 (2013) 126–135

Contents lists available at ScienceDirect

Ultramicroscopy

journal homepage: www.elsevier.com/locate/ultramic

Assessing the precision of strain measurements using electron backscatter diffraction – part 1: Detector assessment

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ARTICLE INFO

Article history:
Received 12 June 2013
Received in revised form
8 August 2013
Accepted 8 August 2013
Available online 21 August 2013

Keywords:
EBSD
Strain
Cross-correlation
Silicon
Deformation

ABSTRACT

We analyse the link between precision of pattern shift measurements and the resolution of the measurement of elastic strain and lattice rotation using high resolution electron backscatter diffraction (HR-EBSD). This study combines analysis of high quality experimentally obtained diffraction patterns from single crystal silicon; high quality dynamical simulations using Bloch wave theory; quantitative measurements of the detector Modulation Transfer Function (MTF) and a numerical model. We have found that increases in exposure time, when 1×1 binning is selected, are the primary reason for the observed increase in sensitivity at greater than 2×2 binning and therefore use of software integration and high bit depth images enables a significant increase in strain resolution. This has been confirmed using simulated diffraction patterns which provide evidence that the ultimate theoretical resolution of the cross correlation based EBSD strain measurement technique with a 1000×1000 pixel image could be as low as 4.2×10^{-7} in strain based on a shift precision of 0.001 pixels.

1. Introduction

The high angular resolution electron backscatter diffraction (EBSD) technique has emerged as a method that enables precise measurement of elastic strain and lattice rotation in bulk samples using a conventional scanning electron microscope (SEM). This method is based on previous work due to Troost et al. [1] and Wilkinson [2] who reported that following the movement of zone axes between a diffraction pattern captured from a deformed and undeformed area of a crystalline sample enabled measurement of the deviatoric strain and lattice rotation tensors. Wilkinson, Meaden and Dingley (WMD) expanded this work by introducing both a full mathematical framework relating shifts to strain and lattice rotation, as well as an implementation of a cross correlation scheme which measures pattern shifts with sub-pixel precision and therefore improves strain sensitivity, with a reported precision of $\sim 10^{-4}$ rads and $\sim 10^{-4}$ elastic strain [3]. In the same year a related paper was published, which included the use of Hooke's law with the traction free boundary condition to separate normal strain components (as a volumetric expansion does not induced any change in interplanar angles) [4]. Subsequently, Britton and Wilkinson presented the use of a robust statistics to improve the measurement range from $\pm 8^{\circ}$ to $\pm 11^{\circ}$ for lattice rotation [5] and importantly this work highlighted problems in measuring elastic strain in the presence of large lattice rotation. These problems have been resolved recently, using an approach that involves image remapping to account for lattice rotation and therefore to measure elastic strain with high precision, even in the presence of large lattice rotations [6,7].

Quantifying resolution of the technique is difficult and for clarity, we initially define terms that are commonly used to describe resolution; notably 'accuracy', 'precision' and 'sensitivity'. We define accuracy as the difference between the correct answer and a result recorded by measurement. In practice, it is difficult to describe an experiment where the correct result is known exactly and as a consequence, fundamental tests of accuracy using experimental patterns are difficult to find. Hence, it is generally easier to describe and measure the precision of a measurement which describes the ability to measure a similar answer, generally for multiple similar measurements. Logically it is therefore possible to be precise but ultimately not accurate (i.e. an inappropriate test can convincingly measure the same solution and therefore be precise, but a fundamental and potentially unknown flaw could render the measurement inaccurate). Practically it is often difficult to ascertain the difference between accuracy and precision in a measurement. In these cases it is simpler to refer to the sensitivity of the method as a more generic, but less satisfying, term.

For cross correlation based high resolution EBSD the analysis route involves several simple steps that have been combined. This hinders direct analysis of experimental errors. Several groups have

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http://dx.doi.org/10.1016/j.ultramic.2013.08.005

approached this problem using experimental data, numerical calculations and simulated patterns which have resulted in different metrics that aim to describe sensitivity. Note that these studies have been largely based upon semiconductor samples, primarily due to the ease with which high quality diffraction patterns can be obtained.

Wilkinson et al. have reported a similar measurement of lattice rotation for a crystal rotated within the microscope, where the resolution was evaluated by measuring the same rotation of a silicon sample using a stage movement [3]. A precision of $\sim 1.5 \times 10^{-4}$ rad was reported, derived from the standard deviation of several patterns captured for each sample rotation using twenty 256×256 pixel region of interest (ROI) with high quality 1000×1000 pixel patterns. In a second study, the test was repeated with a GaAs crystal [8] and from zero rotation to a nominal rotation of 5° the standard deviation increased from 5×10^{-5} to 1.5×10^{-4}. Furthermore a simple beam shift experiment was performed, where in effect a movement in pattern centre results in a shift of the pattern and for twenty ROI a typical standard deviation of 0.02 pixels was determined.

Fundamentally, measuring the accuracy of any technique is difficult and in this case it is difficult to identify a sample in which the strain state is suitably arbitrary but also known to a high accuracy as the strain is measured on a local scale ($\sim$20 nm) with a neighbouring strain free reference. Furthermore, the following variables must be considered in designing an appropriate calibration experiment: determination of pattern centre location, and measurement of detector distortions.

Vaudin and co-workers have measured the stress, strain and surface displacements in a line scan perpendicular to wedge indents in silicon using EBSD and validated these directly with Raman and atomic force microscopy (AFM) [9,10]. In the Raman study, tensile stresses measured using EBSD in the X and Y directions were compared with the effective biaxial stress inferred from the Raman peak shift. Decreasing the wavelength of the Raman laser resulted in a better agreement between HR-EBSD and Raman measurements, which was attributed to differing depths of the interaction volumes for electrons and light, and a significant subsurface stress gradient [9]. In the AFM study, the out of plane components of the displacement gradient tensor measured with HR-EBSD were integrated and compared to topography measurements which gave agreement between the two surface profiles to within 1 nm [10].

Other groups have assessed accuracy using high quality simulated patterns, such as those developed by Winkelmann et al. [11] and Villert et al. [12]. Villert et al. compared the known input strains with those measured using HR-EBSD and found that an accuracy of better than 10^{-4} is possible. In addition error propagation due to uncertainty in the pattern centre position into the measured strain state was measured by inputting the strain state, calculating shifts for each ROI location with a fixed pattern centre; and then back calculating the strain tensor with an inaccurate pattern centre. Errors of 1% in pattern centre position results in errors smaller than 5×10^{-5} for strain. Wilkinson, Meaden and Dingley had made a similar analysis of the effects of pattern centre position on experimental measurements of small lattice rotations [8]. In practice using typical software based calibration routines it is possible to measure the pattern centre within 0.5% of the true solution [13] and therefore accuracy in measuring the pattern centre location is not a limitation on accuracy of the strain measurement, provided that the same camera geometry is used throughout. If patterns have been captured with different camera geometries and compared, then accuracy in the strains is limited by the precision in the determination of the pattern centres which can lead to large artefacts in the measured strains [12,13].

Recently, this technique has been applied to metallic samples [14–16] where there may be significant lattice rotation gradients due to the presence of geometrically necessary dislocations [17]. The combination of large lattice rotations and small strains (near to the elastic limit) can be problematic and results in 'ghosting' of the lattice rotations which are typically noticed as artefacts in the shear strain components [5]. To address this problem, image remapping routines have been applied to measure elastic strain in the presence of large lattice rotations [6,7]. These two approaches differ slightly: the first uses a Hough based orientation measurement to determine the first orientation correction required for remapping and the second uses misorientation derived from a first pass cross correlation measurement. Subsequently the approaches converge, each using a bi-cubic image remapping routine to remove the rotation component from the diffraction pattern such that the elastic strain (and any further small lattice rotations) can be measured more accurately. Using simulated patterns it has been reported that with remapping a 0.5% error in the pattern centre location can result in a strain accuracy of $\sim 5 \times 10^{-4}$ [7]. Therefore advances in pattern centre localisation are predicted to improve the accuracy of this approach significantly.

As this technique has matured, the range of experimental problems to which it has been applied has expanded. Finding the resolution limit and how this varies with exposure time and detector geometry is increasingly important. Especially as many of the current problems under study involve experimentally difficult samples where problems such as charging or carbon contamination, e.g. in III–V nitrides [18,19]; statistically relevant information from large areas [20,21]; or small stresses that lie close to the sensitivity limit (e.g. stresses near cell walls and persistent slip bands in deformed pure metals [22,23]).

In part 1 of this study, we explore and demonstrate factors which affect the precision of the HR-EBSD technique. Initially we study the effect of exposure time and pattern binning on pattern shift precision using line scans of strain free silicon. This in turn has motivated us to characterise the quality of the EBSD detector by measuring modulation transfer function (MTF) which describes its frequency dependent response and links to the ability to measure small image shifts. Subsequently diffraction patterns were simulated and analysed to explore the ultimate accuracy of pattern shift measurements using cross correlation. These shift measurement were linked to strain resolution accuracy through analysis of a simple numerical model.

2. Experiments and simulations

A semiconductor grade silicon single crystal wafer sample was examined in a JEOL JSM-6500F at an accelerating voltage of 20 kV, a nominal probe current of $\sim$10 nA. The sample was tilted to 70°

Table 1
Comparison of binning, image size, intensity ranges and electron budgets (2 s.f.) for silicon diffraction patterns captured with hardware binning.

Hardware binning	1×1	2×2	4×4	8×8
Phosphor diameter (pixels)	904	452	226	113
Acquisition time (s)	1.04	0.24	0.05	0.02
Maximum intensity (arbitrary units)	3550	3450	3400	3550
Minimum intensity (arbitrary units)	1750	1800	1800	1750
Approximate electron budget (electrons per super-pixel)	3900	3600	3000	4700