Ultramicroscopy (xxxx) xxxx–xxxx

Contents lists available at ScienceDirect

Ultramicroscopy

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

Optimizing disk registration algorithms for nanobeam electron diffraction strain mapping

Thomas C. Pekin[a,b], Christoph Gammer[c], Jim Ciston[b], Andrew M. Minor[a,b], Colin Ophus[b,*]

[a] Department of Materials Science and Engineering, University of California, Berkeley, Berkeley, USA 94720
[b] National Center for Electron Microscopy, Molecular Foundry, Lawrence Berkeley National Laboratory, Berkeley, USA 94720
[c] Erich Schmid Institute of Materials Science, Jahnstrasse 12, Leoben, Austria 8700

ARTICLE INFO

Keywords:
Nanobeam electron diffraction
Strain measurement

ABSTRACT

Scanning nanobeam electron diffraction strain mapping is a technique by which the positions of diffracted disks sampled at the nanoscale over a crystalline sample can be used to reconstruct a strain map over a large area. However, it is important that the disk positions are measured accurately, as their positions relative to a reference are directly used to calculate strain. In this study, we compare several correlation methods using both simulated and experimental data in order to directly probe susceptibility to measurement error due to non-uniform diffracted disk illumination structure. We found that prefiltering the diffraction patterns with a Sobel filter before performing cross correlation or performing a square-root magnitude weighted phase correlation returned the best results when inner disk structure was present. We have tested these methods both on simulated datasets, and experimental data from unstrained silicon as well as a twin grain boundary in 304 stainless steel.

1. Introduction

Strain and its spatial distribution is important for a greater understanding of many of the relevant engineering materials currently in use. In most modern silicon devices, strain is an important parameter used to modify the properties of the device itself [1]. Likewise in metallic specimens, understanding strain and its evolution under deformation will help further the understanding and predictive capabilities of the field. While many techniques of measuring strain exist [2–9], scanning convergent nanobeam electron diffraction (NBED) is attractive for a number of reasons. First, NBED strain mapping offers the potential of very high accuracy in strain measurement. Independent diffraction patterns are recorded with high reciprocal space resolution for each probe position, which limits the spatial resolution to the probe size. On a modern scanning transmission electron microscope (STEM), this probe size can easily be below one nanometer while still maintaining a small enough convergence angle to display full diffraction patterns. This can be compared with geometric phase analysis (GPA) strain mapping, in which real space images are acquired with very high spatial resolution, but do not directly sample reciprocal space. Additionally, GPA strain maps are necessarily limited to a small field of view (FOV) as atomic columns must be resolved to make accurate measurements. With NBED this is not a problem and FOV is usually limited by the storage space of the data acquisition system or the sample stability. With the introduction of high speed direct electron detectors, a large number of diffraction patterns can be obtained from a single sample, covering a very large field of view without concerns for sample drift or other instabilities [10,2,11–13].

Cooper et al., have noted that NBED strain measurements can lose accuracy due to non-uniform disk intensity [12]. This non-uniformity is due to experimental limitations such as sample bending, dynamical effects, or imperfect alignment, resulting in more complicated data sets. The effects of dynamical contrast can be dealt with both during and after the experiment. During the experiment, the electron beam can be precessed around the central axis to obtain a radially averaged diffraction pattern. This has been shown to reduce dynamical contrast and return diffraction patterns that can be analyzed as if they were kinematical [14,15]. However, in return for more easily processed diffraction patterns, acquisition speed is slowed to the speed at which the beam can be precessed (~0.1 s per pattern versus 0.0025 s per pattern with a Gatan K2-IS camera), and the spot size increases as microscope aberrations have a larger effect on off axis beams. Alternatively, after the data has been acquired, choices in disk position measurement, lattice fit to disk positions, and image downsampling provide a range of options to optimize the data obtained from malformed disks and recover strain information. Here we present an

* corresponding author.
E-mail address: cophus@gmail.com (C. Ophus).

http://dx.doi.org/10.1016/j.ultramic.2016.12.021
Received 16 August 2016; Received in revised form 8 December 2016; Accepted 9 December 2016

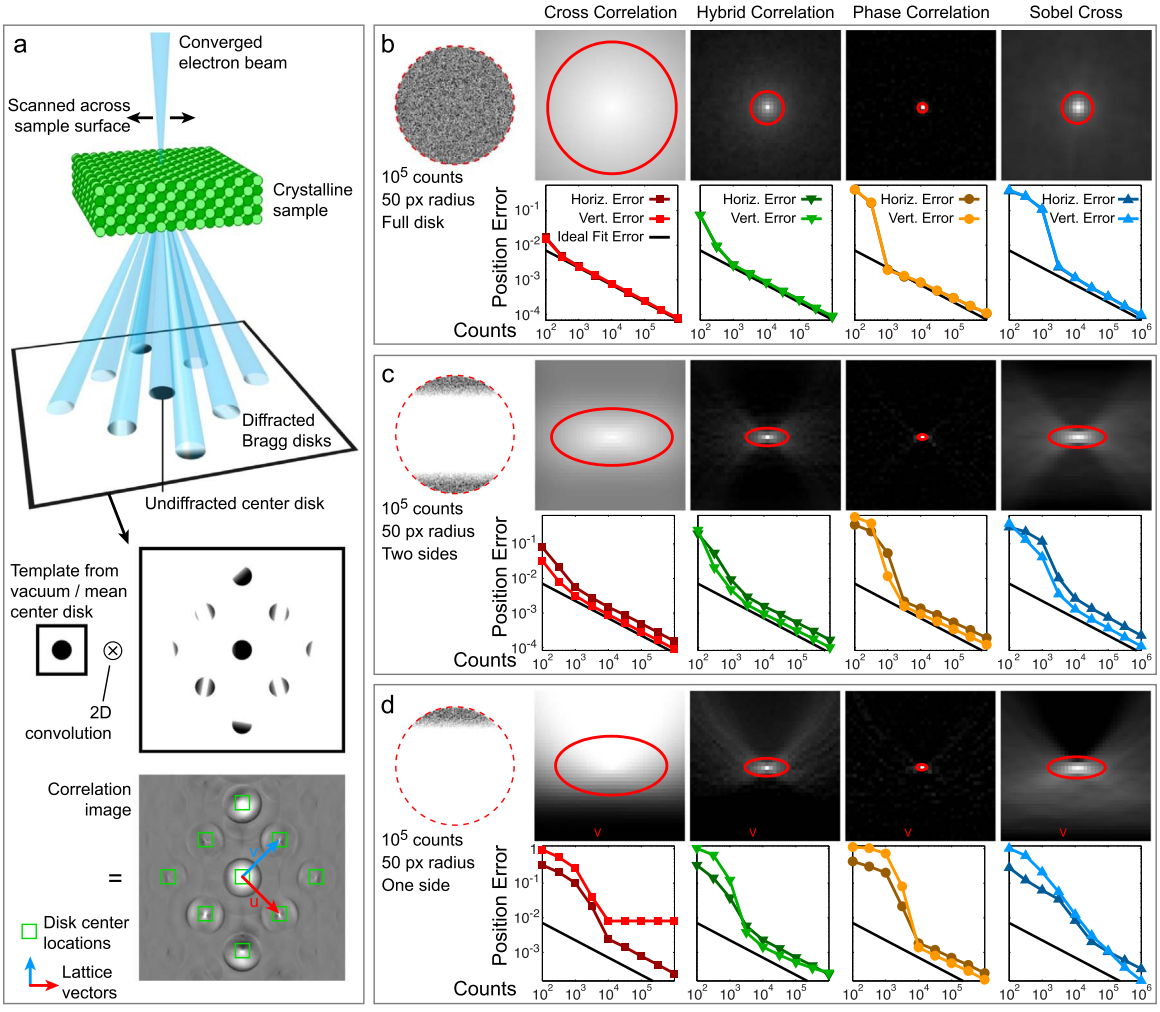

Fig. 1. Strain measurements from nanobeam electron diffraction (NBED). (a) Experimental geometry showing a single NBED measurement. The correlation of the measured diffraction pattern with a center disk template produces an image with sharp peaks at each disk position. Different correlation methods are applied to synthetic disks with (b) no internal structure, (c) disks with signal on opposite edges and (d) disks with signal along a single disk edge. Each method shows an example correlation image, as well as the horizontal and vertical error (divided by disk radius) as a function of counts in the ideal disk without internal structure. Red ellipses show the best fit standard deviations for an elliptic Gaussian function on all peaks. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

overview of the strain mapping technique itself and experimental options, tested on both simulated data as well as relevant experimental results.

2. Theory

2.1. Measuring lattice vectors from nanobeam electron diffraction patterns

Strain measurement via diffracted peak location is a well understood result of Bragg's law, and has been successfully performed using several different experimental techniques [16,10,11]. For NBED, the first and most important step is proper data acquisition. While the sample does not need to be on a perfect zone axis, it must be close enough to have ideally several orders of diffraction disks illuminated. For every pixel in the reconstructed strain image, an entire diffraction pattern must be recorded, shown schematically in Fig. 1a. From each of these patterns, the disk positions are extracted and stored as an (x,y) location in reciprocal space, usually to subpixel precision. After all the disk positions are recorded, they are used to find the local lattice vectors at every probe position. This is done by solving the system of linear equations

$$\mathbf{BL} = \mathbf{P}, \tag{1}$$

for $\mathbf{L}$, where $\mathbf{L}$ is the matrix made up of two lattice vectors defined by pixel lengths from the (000) spot, $\mathbf{P}$ is a matrix of every disk position in pixels, and $\mathbf{B}$ is a matrix of every disk position in normalized lattice vectors. Equivalent rows of $\mathbf{P}$ and $\mathbf{B}$ should correspond to the same diffraction disk for each diffraction disk registered. Often, this calculation is overdetermined, as there ideally will be many more disk positions than lattice vectors. If the solution is overdetermined, the fit accuracy can be improved by using weighted least squares, where the weights are equal to the correlation peak value. This calculation is carried out for every diffraction pattern in the dataset. In addition, a reference lattice $\mathbf{L}_0$ is computed using the disk positions from either a single real space pixel, or the mean of the disk positions for a subset of pixels (the reference region of the dataset).

Once the lattice vectors have been calculated for every diffraction pattern, it is simple to calculate matrix strain using $\mathbf{L}_0\mathbf{T} = \mathbf{L}$, where $\mathbf{L}_0$ is the reference lattice, $\mathbf{T}$ is the transformation matrix, and $\mathbf{L}$ is the current lattice for the pixel in question. If infinitesimal strain theory is assumed, the resulting strain matrix (and infintesimal rotation) is simply

$$\begin{bmatrix} \varepsilon_{xx} & \frac{1}{2}(\varepsilon_{xy} - \theta) \\ \frac{1}{2}(\varepsilon_{yx} + \theta) & \varepsilon_{yy} \end{bmatrix} = \mathbf{T} - \begin{bmatrix} 1 & 0 \\ 0 & 1 \end{bmatrix}, \tag{2}$$