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Strain fields around dislocation arrays in a $\Sigma 9$ silicon bicrystal measured by scanning transmission electron microscopy

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Abstract:

Strain fields around grain boundary dislocations are measured by applying geometric phase analysis on atomic-resolution images obtained from multiple fast acquisitions in scanning transmission electron microscopy. Maps of lattice distortions in silicon introduced by an array of pure edge dislocations located at a $\Sigma 9(122)$ grain boundary are compared with the predictions from isotropic elastic theory, and the atomic structure of dislocation cores is deduced from images displaying all the atomic columns. For strain measurements, reducing the acquisition time is found to significantly decrease the effects of instabilities on the high-resolution images. Contributions from scanning artefacts are also diminished by summing multiple images following a cross-correlation alignment procedure. Combined with the sub-Ångström resolution obtained with an aberration corrector, and the stable dedicated microscope's environment, the rapid-acquisition method provides measurements of atomic displacements with an accuracy below 10 pm. Finally, advantages of combining strain measurements with the collection of various analytical signals in a scanning transmission electron microscope are discussed.

1. Introduction

Strain fields near defects or interfaces not only affect the mechanical properties of materials, but may also significantly modify their electronic properties. In Si-based devices, strain engineering is commonly used to enhance the carrier mobility. Epitaxially grown SiGe in recessed source/drain regions induces a slight deformation of the silicon lattice, resulting in the desired change in conductive properties [1]. In contrast, long range interaction of carriers with defects in silicon often has detrimental effects on device operation, and presents a major challenge in the semiconductor industry. As the conventional scaling in metal-oxide-semiconductor field-effect transistors is no longer sufficient to pursue the continuous miniaturization of devices, new geometries that relies on thin strained silicon channel may present viable alternatives [2]. And considering that the piezoelectric effect in silicon [3] is even larger in nanowires than in the bulk, new applications in nanostructure-based electronics may be envisaged [4]. Moreover, strain-induced electro-optic effects may potentially lead to the design of all-silicon electronic and photonic devices [5]. For a better understanding of the relationship between strain and materials properties, and for a better control on those properties, strain measurements performed at high spatial resolution becomes essential.

Transmission electron microscopy (TEM) has been the only reliable tool to measure strain fields at the nanometer-scale. Several approaches have proved successful, such as convergent beam [6] or nanobeam electron diffraction [7,8], and imaging based on diffraction contrast [9], inline holography [10], or holographic interferometry [11]. Strain maps can also be extracted from atomic-resolution images using peak-finding techniques [12,13,14], or geometric phase analysis (GPA) [15,16,17,18]. With GPA, atomic displacements on the order of 3 picometers were measured using high-resolution TEM (HRTEM), and a quantitative comparison with the elastic theory of dislocations demonstrated the reliability of such measurements. The approach has since been applied to measure strain in the channel of transistors [19] and other semiconductor systems [20,21]. A major challenge in HRTEM-based strain measurements is identifying the errors, introduced by the microscope transfer function, that propagate through the analysis. HRTEM imaging is particularly sensitive to experimental conditions, such as defocus and

specimen thickness, and the observed variations of lattice fringes may not necessarily correspond to real atomic displacements.

Here, we propose to perform geometric phase analysis on images acquired in an aberration-corrected scanning transmission electron microscope (STEM). Annular-dark field (ADF) imaging in STEM produces Z-contrast images [22,23] that are less dependent on experimental parameters and more easily interpretable than HRTEM images. Furthermore, thicker regions of TEM specimens can be analyzed with ADF-STEM. Since strain relaxation occurring at the two specimen surfaces modifies the system studied, thicker region are generally more representative of the original bulk sample. GPA in STEM has previously been used to study structural distortions in charge-ordered manganite [24], as well as strain fields in SiGe/Si heterostructures [25,26] and surrounding dislocations [27,28,29]. Because images in STEM are acquired pixel-by-pixel, instabilities and scanning effects introduce distortions that ultimately limit the precision of strain measurements. To reduce these effects, we propose to use rapid acquisitions followed by a cross-correlation and summation procedure. The images are acquired with a sub-Ångström resolution in a STEM equipped with an aberration corrector. With this approach, strain fields surrounding arrays of dislocations located at a $\Sigma 9$ grain boundary in silicon are mapped. The $\Sigma 9(122)$ system has been studied extensively by Jany Thibault and her colleagues as a model sample to analyze atomic mechanisms occurring at a grain boundary [30]. It has been part of a larger study on interaction between dislocations and grain boundaries [31,32,33,34,35,36]. In the present work, the precision of strain measurements around grain boundary dislocations is discussed, and the results are quantitatively compared to calculations based on the elastic theory.

2. Geometric Phase Analysis in Scanning Transmission Electron Microscopy

Geometrical phase analysis measures atomic displacements in high-resolution images by combining real-space and Fourier-space information. As described in Reference 15, a small aperture is centered on a strong reflection in the Fourier transform of an atomic-resolution image. By extracting the phase component of the inverse Fourier transform of the selected region, a real-space map of the local atomic displacements associated with the strong reflection can be

calculated. Two-dimensional displacement fields are obtained by selecting two non-collinear reflections in Fourier space. The precision of this approach in HRTEM can be more than 100 times higher than the microscope's resolution. All the components of the strain field tensor are calculated from derivatives of the displacement fields. Thus, strain maps obtained from GPA are particularly sensitive to fluctuations, and this presents a challenge for images acquired in a scanning mode.

In scanning transmission electron microscopy, an electron probe is scanned over a specimen in a raster pattern, and scattered electrons are detected [23]. An image is formed by plotting the scattered electron intensities as a function of the probe position. Typically, imaging is performed with a bright-field (BF) detector or an annular dark-field (ADF) detector, both located in the far-field at the diffraction plane. Other signals can also be collected as the electron probe is scanned, such as diffraction patterns or analytical signals from electron energy-loss spectroscopy (EELS) or energy dispersive x-ray spectroscopy (EDS). The ADF imaging mode is unique to STEM. In this mode, the detector collects electrons scattered at high angles over an annulus on the diffraction plane. By setting a high inner collection angle, ADF imaging provides a contrast sensitive to the atomic number Z , resulting in atomic resolution images that are easier to interpret in terms of atomic structure than HRTEM images.

Because STEM relies on a serial acquisition, as opposed to a parallel acquisition as in conventional TEM, instabilities may introduce distortions in the images. We divide these instabilities in two categories. The first category is the so-called “flyback error”, which consists of a beam shift between scan lines. If we consider x as the scan direction, when the scan reaches the end of a line at (x_{end}, y_i) , instead of going back exactly to the next line at (x_0, y_i+1) , the probe will instead start at a position displaced by $\Delta_F(y)$. As described in Ref. [25], this flyback error $\Delta_F(y)$ depends only on the variable y , i.e. the component perpendicular to the scan direction. It can be separated into a x and a y displacement, $\Delta_{Fx}(y)$ and $\Delta_{Fy}(y)$ respectively. The second category includes all other instabilities, arising for instance from electromagnetic fields, sample drift, vibrations, or other environmental noise. In this case the displacement $\Delta_I(x,y)$ will depend on both x and y , and can also be separated in a x and y component, $\Delta_{Ix}(x,y)$ and $\Delta_{Iy}(x,y)$.

respectively. By considering the local atomic displacement along x and y , $u_x(x,y)$ and $u_y(x,y)$, the strain tensor ε that is measured experimentally ($[\varepsilon]_{exp}$) in STEM can be written as follow:

Equation 1

$$\begin{aligned} [\varepsilon_{xx}]_{exp} &= \frac{\partial(u_x(x,y) + \Delta_{fx}(x,y))}{\partial x} \\ [\varepsilon_{yy}]_{exp} &= \frac{\partial(u_y(x,y) + \Delta_{fy}(x,y) + \Delta_{Fy}(y))}{\partial y} \\ [\varepsilon_{xy}]_{exp} &= \frac{1}{2} \left(\frac{\partial(u_x(x,y) + \Delta_{fx}(x,y) + \Delta_{Fy}(y))}{\partial y} + \frac{\partial(u_y(x,y) + \Delta_{fy}(x,y))}{\partial x} \right) \end{aligned}$$

The flyback Δ_F error depends only on y , and therefore appears only for the ε_{xy} and ε_{yy} components of the strain tensor. As explained in Ref. [25], its effect on strain measurement is therefore limited to a direction perpendicular to the scan direction. For a scan direction x , ε_{xx} measurements will be significantly more accurate than ε_{yy} and ε_{xy} measurements. Thus, previous GPA-STEM results have usually displayed only ε_{xx} maps. However, the other source of instability $\Delta_l(x,y)$ will affect all the components of the strain tensors.

In the present work, atomic resolution images used for GPA are obtained through a sum of multiple fast acquisitions. The acquisition time was lowered to 2 s for an image, around the same acquisition time than for a typical HRTEM image, and around 30 times faster than for a typical STEM image. By rapidly collecting the images, the influence of low-frequency components of Δ_l will be reduced. The sum of multiple images provides a suitable signal to noise ratio for GPA, and also averages the effects of the flyback error Δ_F and the high-frequency components of Δ_l .

3. Experimental Methods

3.1 Electron Microscopy

Scanning transmission electron microscopy experiments were carried out at the Canadian Center for Electron Microscopy on a FEI Titan 80-300 “cubed” fitted with an aberration-corrector for both the probe-forming lens system and for the imaging lens system. The microscope is equipped with a high-brightness field emission source (X-FEG) and a monochromator. The accelerating voltage can be varied between 80 kV and 300kV. It was set to 200 kV for the experiments presented here. The stable dedicated environment of the microscope plays a crucial role in reducing the instabilities that could affect strain measurements. The convergence semiangle was set at 24 mrad, and the collection semiangle of a Fischione high-angle annular dark-field (HAADF) detector was set at 60 mrad.

Plane-view TEM specimens were prepared from a deformed $\Sigma 9$ bicrystal sample described in the next sub-section. The specimens were prepared by the tripod-mechanical polishing method using a MultiprepTM. The specimens were polished with a wedge angle of 2 degrees until electron transparency and were then mounted on a molybdenum window grid. As a final cleaning step, the specimens were ion milled at low energy (250eV) for a short time (15 minutes) with a Technoorg Gentle MillTM. The specimens were oriented along the [011] zone axis. Some bending of the silicon was observed on the diffraction pattern for the thinnest part of the specimen. To minimize the effect of surface relaxation on the GPA strain measurements, atomic resolution images were taken in thicker regions of the specimens, following an inspection of diffraction patterns to ensure that the grain boundary analyzed consisted only of a rotation of two silicon crystals along the [110] axis as described below.

3.2 Arrays of pure edge dislocations located at a $\Sigma 9(122)$ grain boundary in silicon

A pure tilt silicon bicrystal, containing initially a $\Sigma 9(122)$ grain boundary, was deformed in compression at a temperature of 1200°C. The interface of the two crystals following deformation has previously been analyzed in detailed studies using HRTEM [30,35,36]. As described in Ref. [30], the predominant deformation-induced 60° dislocations decompose into grain boundary dislocations. At high temperature, one type of dislocation with a burgers vector b_c remains absorbed at the boundary, and is often distributed in an array, forming a sub-grain superimposed

on the original $\Sigma 9$ boundary that increases slightly the misorientation angle between the two grains.

The bicrystal was grown by the Czochralski pulling method, and contains a twist boundary that separates two perfect crystals rotated with respect to each other by an angle of 38.94° along the common $[011]$ axis. Figure 1(a) shows an ADF-STEM image of the bicrystal. The vector perpendicular to the plane of the grain boundary is $[12\bar{2}]$, expressed with respect to the silicon lattice on the right of the boundary. The atomic structure of the $\Sigma 9$ boundary, which has first been elucidated in germanium [37], can be described by two structural units consisting of 7 and 5 atoms, producing a periodic vector of $a/2[\bar{4}\bar{1}1]$ (where a is the lattice parameter of Si), as shown in the inset of Fig. 1(a). The structural units appear clearly in the inset of Figure 1(a), and the periodic vector is also shown. In this work, we focus on three parts of the deformed bicrystal sample: (1) regions without defects (2) regions with arrays of edge dislocations, and (3) regions with isolated dislocations. Figure 1 present two images taken on TEM specimens prepared from the same sample. The defects visible in figure 1(b) are edge dislocations with Burgers vector $a/9[12\bar{2}]$, which has been determined using the method of King and Smith [38]. Each dislocation introduces a structural unit consisting of 6 atoms. In the inset of figure 1(b), the center of this unit is labeled.

3.3 Image Acquisition, Processing and Analysis

Figure 2 presents an overview of the steps in the procedure. First multiple images are acquired with a low pixel dwell time (Fig. 2(a)). In the example shown here, 13 images were acquired with a sampling of 0.0162 nm and a dwell time of 0.4 μ s giving an acquisition time of 2s for each image. The images are then aligned through a cross-correlation procedure, and summed. The result is an image with a good signal to noise ratio (Fig. 2(b)). It displays a perfect $\Sigma 9$ grain boundary except for a region near the top that contains an isolated edge dislocation, as shown in the inset. The scanning direction was chosen to be normal to the boundary plane, which corresponds to the burgers vector direction of the edge dislocation (in figure 2, it corresponds to the horizontal direction).

On the diffractogram of this image (Fig. 2(c)), reflection spots for the two Si crystals on both side of the grain boundary are observed. The geometric phase analysis was performed using the FRWRtools plugin, which is a script implemented in the DigitalMicrograph (Gatan) environment by Christoph Koch, and is based on the geometric phase analysis developed by Hytch, Snoeck, and Kilaas [15]. In the present work, two non-collinear (111) reflections were selected from the diffractogram to obtain the strain maps. The spatial resolution of strain maps is inversely proportional to the size of the aperture used to select reflections in Fourier space. To avoid artifacts arising from a sharp cutoff in Fourier space, the edge of the circular aperture were smoothed by a cosine function. With a larger mask, the resolution would be improved and it might also be possible to include reflection spots from both crystals. This approach has been used for grain boundaries with lower misorientation angles [15]. However, selecting a larger mask would also introduce more noise in the phase maps. For all the results presented here, a resolution of 2.5 nm was selected.

Figure 2(d) displays the phase images corresponding to the (111) reflections of the Si crystal on the right of the grain boundary. To obtain these maps, an additional refinement step was used. It consists in minimizing the phase within a region of undistorted Si away from the boundary. Two well-defined regions are observed on the phase images. On the left, the phase varies randomly. This is because the reflections selected correspond to the Si lattice on the right of the grain boundary. On the right of the phase image, the signal varies slowly, except for the region close to the isolated dislocation. As explained in Ref. [15], the phase can be related to the local atomic displacements, from which the strain fields can be deduced from an analysis of the derivatives. The strain components ϵ_{xx} , ϵ_{yy} and ϵ_{xy} are shown in Figure 2(e). As a comparison, Figure 2(f) shows the strain fields for the same specimen region obtained from a single STEM acquisition. The improvement of the method proposed here is particularly evident for strain tensors with a y-component.

4. Results

Strain maps for the $\Sigma 9(122)$ grain boundary with an isolated edge dislocation are shown in Fig. 3. The GPA was performed separately for the lattices on the right and the left of the grain boundary, and the strain maps were joined together. No strain fields associated with the grain boundary were detected in this analysis. Since the $\Sigma 9$ structure is a low energy configuration, its associated long-range distortions of the Si crystals are expected to be relatively small (i.e. below our detection limit). It should be mentioned that the presence of the grain boundary is also expected to influence the strain fields surrounding dislocations [17]. This effect, as discussed in the next section, is also below our detection limit. Comparison with the elastic theory will therefore be done for the case of an isolated dislocation in a perfect silicon lattice.

Maps calculated from a linear isotropic elastic model describing the strain fields surrounding an individual edge dislocation with a Burgers vector of $a/9[12\bar{2}]$ are presented in figure 3. The stress tensor σ_{ij} for an edge dislocation was taken from reference [39]. Assuming a linear relationship between stress and strain, the tensor components for the strain fields can then be obtained from Hooke's law. And because the dislocation and the grain boundary are uniform along $[011]$, the plane strain condition with $\varepsilon_{zz} = 0$ applies. For that geometry, σ_{xz} and σ_{yz} are both equal to zero, and $\sigma_{zz} = \nu(\sigma_{xx} + \sigma_{yy})$ where ν is Poisson's ratio (0.218 for silicon). The isotropic stress-strain relation can then be written as:

Equation 2

$$\begin{aligned}\sigma_{xx} &= 2\mu\varepsilon_{xx} + \lambda(\varepsilon_{xx} + \varepsilon_{yy}) \\ \sigma_{yy} &= 2\mu\varepsilon_{yy} + \lambda(\varepsilon_{xx} + \varepsilon_{yy}) \\ \sigma_{xy} &= 2\mu\varepsilon_{xy}\end{aligned}$$

Where μ and λ are the shear modulus and the Lamé constant. For silicon, $\mu = 0.681$, and $\lambda = 0.524$. Although silicon is anisotropic, the isotropic theory proves to be sufficient in the present analysis. It is also important to note that the theory is no longer accurate in the immediate vicinity of a dislocation core, where a non-linear atomistic theory would be required. Generally, the elastic theory is accurate for distances of more than ~ 1 nm from a dislocation core

From the comparison presented in Figure 3, it is clear that the experimental results reproduce well the expected pattern for the ε_{xx} , ε_{yy} and ε_{xy} strain tensor components. Furthermore, as will

be discussed below in more details, the intensity scale is similar for both the measured and the predicted strain maps, except close to the dislocation core.

For the case of an array of edge dislocations located at the grain boundary, the experimental and theoretical results are shown in Figure 4. All dislocations have a Burgers vector of $a/9[12\bar{2}]$ in a direction parallel to the normal of the $\Sigma 9$ boundary plane. The strain maps are obtained following the same procedure described in section 3, with a resolution for the analysis also set at 2.5 nm. The theoretical strain maps are obtained using the same isotropic theory transformation of equation 2 with a stress tensor taken from Ref. [39]. The patterns appear qualitatively similar than their counterparts for an isolated dislocation. However, noticeable differences are both expected and detected, as will be discussed below. Again the experimental results reproduce well the predicted ones for regions away from the dislocation cores, with the exception of the ϵ_{yy} map. The predicted three-fold pattern is reproduced in the experimental map only for the dislocation near the bottom of the image, and not for the two dislocations above.

In Figure 5, ϵ_{xx} profiles taken along a line parallel to the grain boundary, along the $[\bar{4}\bar{1}1]$ direction, are shown for both an isolated dislocation and an array of dislocations. The profiles were taken 2.5 nm away from the boundary. Experimental results are shown in (a) and theoretical results in (b). In both cases, the measured amplitude is close to the predicted value. For the isolated dislocation, the experimental results appear to be shifted upward, whereas the model predicts a centered profile. As we have seen in Fig. 2(d), the phase slowly vary over distances of a few nanometers. Thus, a vertical shift in strain profiles may be introduced because the area selected for the refinement step (where the phase is minimized) is away from the grain boundary. Overall, the behavior predicted by the elastic model is quantitatively reproduced by the experiments. The ϵ_{xx} strain component for the dislocation array displays a profile with a lower amplitude and a faster decay due to the presence of neighboring dislocations.

5. Discussion

The precision of strain measurements from geometric phase analysis depends on the quality of the image, but also on contributions from possible artifacts. When artifacts arising from the imaging system or a deformation of the specimen are present, the experimental images are no longer representative of the system studied. The possible sources of artifacts that are characteristic to STEM are described in section 2, and include the flyback error and other sources of instabilities arising from sample drift, electromagnetic interference, or vibration. Concerning the image quality, the signal-to-noise ratio, the spatial resolution, and the sampling will determine the noise level in the phase maps. In the present experiments, the images were collected in an aberration-corrected STEM with a sub-Å resolution, and a signal-to-noise ratio suitable for GPA was obtained by summing multiple images following a cross-correlating procedure. Reducing the phase noise is also possible by using a smaller mask to select the reflections in the diffractogram, however this is done at the expense of spatial resolution in the phase maps. A mask corresponding to a spatial resolution of 2.5 nm was found to be optimal for the present experiments.

To quantify the level of precision reached with the proposed STEM-based method, figure 6 displays an analysis of measured atomic displacements for a region of perfect Si crystal. Phase maps were calculated by performing the analysis described in section 2. As explained in Ref. [15], the atomic displacement u can be obtained from the phase maps P for the selected reflection g_1 and g_2 with

Equation 3

$$\begin{pmatrix} u_x \\ u_y \end{pmatrix} = -\frac{1}{2\pi} \begin{pmatrix} g_{1x} & g_{1y} \\ g_{2x} & g_{2y} \end{pmatrix}^{-1} \begin{pmatrix} P_{g_1} \\ P_{g_2} \end{pmatrix}$$

Where P_{g_1} and P_{g_2} are the phase maps for the reflection $g_1 = (\bar{1} \ 1 \ \bar{1})$ and $g_2 = (11 \ \bar{1})$, and g_{1x} (g_{2x}) and g_{1y} (g_{2y}) are projection of g_1 (g_2) along the x and y axis. Here, the scanning direction is in the x-direction ($[12\bar{2}]$), and the y-axis runs along the grain boundary in the $[\bar{4}1 \ \bar{1}]$ direction. The displacement maps are shown in Figure 6. The standard deviation of the total displacement is 0.05 Å. Since strain fields are derivatives of atomic displacements, their measurements will be strongly affected by phase fluctuations. Furthermore, strain fields extracted from GPA are only

accurate within a limited region surrounding the dislocations. For positions too close to the dislocation cores, the assumption of a continuous displacement field is no longer valid. And for measurements taken too far from the core, the noise level becomes dominant.

As described in Ref. [17], for a $\Sigma 3(111)$ coherent twin boundary in germanium, evidence that a grain boundary have different elastic moduli than the single crystal matrix can be found by analyzing the strain fields variations near dislocations located at that boundary. For the $\Sigma 9$ grain boundary in silicon, the precision obtained in our experiments was too low to detect the influence of the grain boundary on the strain fields. Since this bicrystal represents a low energy configuration, we expect this influence to be small and therefore hard to detect. Overall, the experimental results reproduced well the strain fields predicted by a linear isotropic elastic theory for edge dislocations in a single crystal matrix, except for some ϵ_{yy} maps that did not always reproduce the expected pattern. Since decreasing the acquisition time does not remove the flyback error, the effect on strain maps with a y -component of summing multiple images is not intuitive. When practical, a solution would be to reproduce the multiple rapid acquisition method for all the axes of interest for a system. For a straight grain boundary with dislocations having a Burgers vector perpendicular to the boundary, two perpendicular scanning directions would be sufficient for a complete analysis.

Figure 7 presents a GPA analysis for the same system (i.e. array of edge dislocations located at a $\Sigma 9$ grain boundary in silicon) on a high-resolution TEM image. No scanning effects are expected from GPA maps extracted from a HRTEM image, since the acquisition is done using a CCD camera rather than by scanning an electron beam. The HRTEM was acquired at 200 keV with an aberration-corrector for the imaging lens, which provides a spatial-resolution comparable to STEM. The acquisition time is 1s, and the sampling is 0.0266 nm. The image shown in Fig. 7 was taken from a thick region of the specimen, like the previous ADF-STEM images, to minimize the influence of surface relaxation. Because of thickness effects, HRTEM images do not display a contrast directly representative of the atomic structure [40]. For instance, the Si dumbbells are not visible, and the contrast near the dislocations varies strongly. The ϵ_{xx} and ϵ_{xy} maps qualitatively reproduce the results predicted by the elastic theory, however the expected ϵ_{yy} pattern is not observed. This suggests that the difficulties in reproducing the ϵ_{yy} pattern in Figure

4 are not solely due to scanning effects. The ε_{yy} strain component may be harder to measure for this system, or may be influence by the presence of the $\Sigma 9$ boundary. A thorough comparison between STEM and HRTEM for strain analysis is beyond the scope of this work, and would need to include all the acquisition parameters and conditions. For instance, the lower precision displayed in Fig. 8 for the ε_{xx} (when compared to Fig. 4) may be due to a difference in the specimen regions analyzed.

Performing GPA on STEM images present several advantages. Since the contrast in ADF imaging is less affected by the specimen thickness, strain measurements can be performed for thicker regions that are less affected by strain relaxation at the specimen surfaces. ADF-STEM images also provide a Z-contrast, rendering the interpretation of images more intuitive. Figure 6 presents an ADF image of a dislocation array overlapped with the strain ε_{xx} and the rigid-body rotation ω_{xy} . The later involves derivatives in a direction parallel and perpendicular to the scanning directions, and is defined as

Equation 4

$$\omega_{xy} = \frac{1}{2} \left(\frac{\partial u_x}{\partial y} - \frac{\partial u_y}{\partial x} \right)$$

As the images in Fig. 8 demonstrate, when performing GPA in STEM, it is possible to obtain simultaneously the exact structure of dislocation cores, with all the atomic columns visible, and the strain fields surrounding those dislocations. For the region in the vicinity of the dislocation cores, where the elastic theory is expected to fail, the position of the atomic columns could also be compared with predictions from atomistic theory, and thus provide a more complete picture.

STEM also offer the possibility to acquire diffraction patterns or spectroscopy signals with the same electron probe used to obtain the images. Diffraction patterns also contain information about strain. Furthermore, electron diffraction can detect small rotation of crystals along axes perpendicular to the optical axis, whether arising from the grain boundary structure or from deformation of the specimen due to strain relaxation. In the current experiment, variations in the diffraction patterns across the grain boundary were observed to ensure the region analyzed did not display significant specimen deformation. Furthermore, information about dislocation core

reconstruction is contained in the diffraction pattern [41]. Another advantage of performing GPA in STEM is the possibility to combine strain measurement with the mapping of the chemical composition. This information can be extracted from Z-contrast STEM images, often with single-atom sensitivity [42,43], and may be used to study the influence of strain fields on the distribution of dopant atoms. Spectroscopic techniques such as electron energy-loss spectroscopy (EELS) or energy dispersive x-ray spectroscopy (EDS) also provides elemental maps with atomic-resolution [44,45,46]. And with the continuous increase in energy resolution, it might also become possible to probe directly with EELS the electronic structure near dislocation cores [47].

6. Conclusions

Strain mapping around dislocation arrays located at a $\Sigma 9$ grain boundary in silicon were obtained through geometric phase analysis of annular dark-field (ADF) images acquired in aberration-corrected scanning transmission electron microscopy (STEM). To reduce the influence of scanning effects and instabilities, the analysis was performed on series of rapidly acquired ADF-STEM images. By increasing the acquisition rate, a sub-10pm accuracy in the measurement of atomic displacements was reached, and by summing multiple images following a cross-correlation alignment procedure, acceptable signal-to-noise ratios were obtained and scanning effects were reduced. Maps of all the strain tensor components in the imaging plane could then be directly overlaid with images of dislocations displaying all the atomic columns, combining a direct atomic structure determination of the defects with a precise measurement of lattice distortions surrounding them. The method proposed here also presents additional advantages over analyzes based on conventional high-resolution transmission electron microscopy, as electron diffraction patterns and analytical signals can be collected with the same electron probe used for ADF-STEM imaging. This will potentially provide more detailed analyses, from mapping the chemical composition to probing the electronic structures, and may lead to a better control of strain-dependent properties on the nanometer scale.

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Figure 1. (a) ADF-STEM image of a perfect $\Sigma 9(122)$ grain boundary in silicon viewed along the $[011]$ zone axis. The orientation vectors are given with respect to the silicon grain on the right. The inset shows a close-up of the grain boundary, and its periodicity vector of $a/2[\bar{4}11]$ is indicated by an arrow. (b) ADF-STEM image of an array of edge dislocations located at the $\Sigma 9$ grain boundary. The inset on the left shows a close-up of a dislocation core, and the inset on the right shows a low magnification image of the dislocation array. The images were acquired from a single acquisition (frame time ~ 60 s), and were smoothed with a Butterworth filter.

Figure 2. Steps taken to obtain strain maps. (a) N images obtained from rapid acquisition are aligned with a cross-correlation procedure and summed. (b) The resulting image displays a $\Sigma 9$ grain boundary containing a single b_c dislocation, as shown in the inset image on the right. On the diffractogram shown in (c), two reflection spots are selected to calculate the phase image shown in (d). (e) From a derivative analysis, ϵ_{xx} , ϵ_{yy} and ϵ_{xy} maps are extracted. As a comparison, the results of the same analysis performed on a typical STEM image of the same region obtained from a single acquisition are shown in (f).

Figure 3. Strain maps for an isolated dislocation located at the $\Sigma 9$ grain boundary. The experimental results (top) were obtained from the ADF-STEM image on the left, and the modeled maps (bottom) were derived from a linear elastic theory, as described in the text. The ADF-STEM image was obtained from 13 images with 2048x2048 pixels (binned by 2 for the analysis) acquired with a frame time of 2s and a sampling of 0.013 nm.

Figure 4. In-plane strain tensor components for an array of dislocations located at the $\Sigma 9$ grain boundary. The experimental results (top) were obtained from the ADF-STEM image on the left, and the modeled maps (bottom) were derived from a linear elastic theory for a dislocation array in a single crystal. The ADF-STEM image was obtained from 23 images with 2048x2048 pixels (binned by 2 for the analysis) acquired with a frame time of 2s and a sampling of 0.013 nm.

Figure 5. Local ϵ_{xx} strain profile measured experimentally (a), from the results shown in Fig. 3 and 4, and determined from the elastic theory (b). The profiles are taken in a direction parallel to the grain boundary along $[\bar{4} \bar{1} 1]$ at a distance of 2.5 nm from the boundary. The dotted lines (blue) display the results for the isolated dislocation, and the solid lines (red) display the results for the array of dislocations.

Figure 6 ADF-STEM image (left) of silicon, with the displacement fields (right) along the x and y directions. The image has been obtained from 15 images with 2048x2048 pixels (binned by 2 for the analysis) acquired with an acquisition time of 2s and a sampling of 0.013 nm.

Figure 7. In-plane strain tensor components for an array of dislocations located at the $\Sigma 9$ grain boundary. The maps were obtained from the HRTEM image on the left. The HRTEM image was acquired with 2048x2048 pixels (binned by 2 for the analysis), a frame time of 1s and a sampling of 0.026 nm.

Figure 8. Atomic-resolution ADF-STEM image of the array of dislocations located at the grain boundary overlapped with the maps of the strain component ϵ_{xx} (left) and of the rigid-body rotation ω_{xy} (right).

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