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A Tilting Procedure to Optimize Energy-Filtered TEM Imaging of Planar Interfaces

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Abstract It is demonstrated that energy-filtered transmission electron microscope imaging of a planar interface between two crystals can be optimized by orienting a sample so that the interface is parallel to the electron beam, but not directly on a zone axis. This orientation reduces diffraction contrast in the unfiltered (and zero-loss) image, which in turn, reduces residual diffraction contrast in raw energy-filtered images (EFI), jump-ratio images and elemental maps. This tilting procedure produces EFI which are more directly interpretable and, in many cases, possess superior spatial resolution and compositional contrast compared to EFI acquired directly on a zone axis.

Keywords: energy-filtered transmission electron microscopy (EFTEM), electron energy-loss spectroscopy (EELS), signal-to-noise ratio (SNR), energy-filtered image (EFI), elemental map, jump-ratio image, **semiconductor interfaces**

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Introduction

The study of the crystal structure and chemical composition of interfaces is an important aspect of metallurgy, materials science and mineralogy [1]. While the structure of an interface can be imaged by high-resolution transmission electron microscopy (HRTEM) or high-angle annular dark-field imaging (HAADF)¹, direct chemical information must be obtained by techniques such as energy dispersive X-ray spectroscopy (EDXS), electron energy-loss spectroscopy (EELS) or energy-filtered transmission electron microscopy (EFTEM). Both X-ray and EELS data can be collected in a conventional TEM (CTEM) or in a scanning transmission electron microscope (STEM). When a CTEM is used in conjunction with an energy filter to extract chemical information from a crystalline specimen, the detrimental effects of diffraction contrast can often be seen in energy-filtered images (EFI) as residual diffraction contrast [2-15]. This preservation of diffraction contrast in EFI makes image interpretation more difficult and less reliable since intensity fluctuation in the EFI are caused not only by compositional gradation, but also by dynamical **diffraction** processes (i.e., diffraction contrast).

In TEM studies of **studies** interfaces, a zone axis orientation is usually chosen in order to align the interface exactly parallel to the electron beam. When oriented on a zone axis, multiple beams are excited and if a moderately sized objective aperture is used (say a collection angle β of 10 mrad), substantial, unwanted diffraction contrast can occur. Alternatively, if the interface is

¹ Chemical information can be extracted from HAADF images, however, this information is derived from the relative intensity of one atomic column compared to another and is not quantitative.

planar, the sample can be tilted into an orientation where the interface is still parallel to the electron beam, but most intensity is in the 000 beam and diffraction contrast is markedly reduced (see Figure 1 for example). This is achieved by tilting the sample off the exact zone axis orientation along the $\pm hkl$ Kikuchi band that is parallel to the interface. In the case of Figure 1, the sample (sputtered aluminum deposited onto a (001) oriented SIMOX wafer) was tilted along the ± 200 Kikuchi band from the [011] zone axis so that the ± 400 reflections were symmetrically positioned on either side of the 000 beam (optic axis). This causes the (001) interfaces to remain parallel to the beam upon tilting. By minimizing diffraction contrast in a conventional bright-field TEM image, residual diffraction contrast is correspondingly minimized in EFI. The reduction of residual Bragg contrast allows for easier interpretation and quantification of EFI and in many cases, increased spatial resolution.

In this investigation, a SiGe heterostructure with a set of (001) interfaces was used to examine and compare the quality of both jump-ratio images and elemental maps acquired while oriented directly on the [011] zone axis and after tilting off the [011] axis with the (001) interfaces still parallel to the electron beam. Of great interest is to examine how the collection angle (i.e., which reflections are collected) affects the residual Bragg contrast in both jump-ratio images and elemental maps acquired in each orientation.

Sample Preparation and Experimental Procedures

Two samples were used in these experiments. The first structure was 50 nm of sputtered Al deposited onto a commercially prepared (001) oriented SIMOX wafer (consisting of 300 nm

of solid phase epitaxially regrown (SPER) Si on top of 370 nm of implanted SiO₂). The majority of the data presented was obtained from a SiGe multilayer heterostructure grown on a Si (001) substrate. The SiGe heterostructure was composed of a Si substrate with a series of Si_{1-x}Ge_x layers grown so that the germanium content (x) increases from 0 % to 30 % in approximately 5 % increments. Between each SiGe layer there was a small layer (\cong 10 to 15 nm in thickness) of pure Si. Therefore, following the substrate, the first layer of SiGe (25 to 100 nm in thickness) contained 5% Ge, then, following a small layer of pure Si, the second layer (25 to 100 nm in thickness) contained 10% Ge. This sequence proceeded until the final SiGe layer contained 30% Ge.

The samples were prepared for transmission electron microscopy using a standard technique for silicon cross section preparation. Two Si heterostructures were glued together using M-Bond, then placed between two pieces of silicon so that the entire thickness was > 3 mm. The samples were then sliced into 500 μ m pieces using a diamond saw, and the resulting sections were cut into 3 mm discs using an ultrasonic disc cutter. The interfaces of interest were centered in the 3 mm discs. The samples were ~~then~~ ground to 250 μ m by hand, then dimpled to approximated 10 μ m thickness. The samples were ~~then~~ ion milled at room temperature using an argon beam of 6 kV and 1 mA at an angle of 18° from both sides until perforation. A final polishing was performed at 4 kV, 0.5 mA at angle of 14° for an 90 min.

Electron energy-loss spectroscopy and imaging experiments were performed on a Philips CM300 FEG TEM operating at 300 kV and utilizing a Gatan Imaging Filter (GIF) with

EL/P (v. 3.0) and Digital Micrograph (v. 2.5) software [16]. ~~The Si foil was translated to the edge of the hole (where the Ge buffer layers were) and~~ The sample was oriented both on and near the [011] zone axis for analysis. A set of EFI were acquired directly on the [011] zone axis and after tilting 12° along the ± 200 Kikuchi band so that the ± 400 reflections were symmetric about the optic axis (000 beam), assuring that the (001) interfaces were parallel to the beam. Jump-ratio images and elemental maps were acquired in both orientations with several different objective aperture sizes (collection angles ranging from 3 to 16 mrad). Unfiltered and zero-loss images were acquired at a microscope magnification of 17.5 kX, using a 1 sec exposure and 1X binning (1024 x 1024). EFI were acquired at the same magnification with 2X binning (512 x 512) and a 15 eV energy window. Jump-ratio images and elemental maps of the Si $L_{2,3}$ edge (at 99 eV) were acquired using a 20 sec exposure with windows centered at 77, 92 and 125 eV for the pre-edge 1, pre-edge 2 and post-edge images, respectively (Figure 2). EFI were acquired using a 3 mm GIF entrance aperture while EEL spectra were acquired in imaging mode at 0.2 eV/pixel dispersion as the sum of ten 0.5 sec exposures using a 2 mm GIF entrance aperture. Finally, Digital Micrograph [16] was used to obtain, correlate, and divide pre-edge and post-edge images to produce jump-ratio images and elemental maps and to measure the intensities within the images.

Results and Discussion

Figure 3 shows two low-magnification bright-field TEM images of the SiGe heterostructure with the $\text{Si}_{1-x}\text{Ge}_x$ layers at the foil edge. Both images were acquired using an objective aperture with a collection angle $\beta = 3$ mrad, which included only the 000 beam.

Corresponding electron diffraction patterns are shown below each image with a box indicating the area where the patterns were obtained. The box in each bright-field image also represents the area of analysis for all subsequent EFI and EELS (a square was used to represent the area sampled by the charge-couple device (CCD) chip). When the foil was oriented directly on the [011] zone axis, there was a large amount of diffraction contrast in the entire image (and in the box), however, when the sample was tilted along the ± 200 Kikuchi band (labeled as Off [011]) diffraction contrast was markedly reduced. The three white circles superimposed on the diffraction pattern taken directly on the [011] zone axis show the size of and the reflections collected by, each objective aperture used in this experiment. The smallest has a collection angle $\beta = 3$ mrad, the middle has $\beta = 8$ mrad and the largest has $\beta = 16$ mrad.

Figure 4 shows two EFTEM zero-loss images ($\Delta E = 10$ eV) acquired from the boxed area in Figure 3. These images were captured using the CCD camera and an objective aperture with a collection angle of 3 mrad. Again, the diffraction contrast produced when oriented directly on the [011] zone axis is considerably greater than when oriented slightly off the zone axis, but with the interface still parallel to the electron beam.

A set of thickness or t/λ maps, where t = sample thickness and λ = inelastic mean free path [17,18], are presented in Figure 5. A thickness map is created by dividing an unfiltered, bright-field image by a zero-loss image, which normalizes by the unfiltered image. The image, therefore, represents sample thickness variation through the intensity distribution of normalized inelastic scatter, with the intensity increasing with increasing sample thickness.

Immediately noticeable is that the sample is wedge-shaped and is thicker at the top of the image. Additionally, there is a large amount of residual diffraction contrast in the thickness map composed of images acquired directly on the [011] zone axis with $\beta = 3$ mrad. This residual diffraction contrast decreases as the collection angle increases. **Conversely**, there is almost no residual diffraction contrast in the thickness maps acquired off the [011] zone axis independent of collection angle.

Figure 5 also includes a set of numbers representing the relative sample thickness ($\ln(I_T/I_0)$, or $\ln(\text{unfiltered}/\text{zero-loss})$), where I_T is the total scattered intensity (inelastic and elastic) and I_0 is the elastically scattered intensity only. The values of I_T/I_0 were measured directly from the thickness maps using Digital Micrograph and are shown at the tops and bottoms of each image. The absolute sample thickness can be calculated using the equation [17,18]:

$$t/\lambda = \ln\left(\frac{I_T}{I_0}\right), \quad (1)$$

where the inelastic mean free path is given by:

$$\lambda = \frac{106FE_0}{\left\{E_M \ln\left(\frac{2\beta E_0}{E_M}\right)\right\}}, \quad (2)$$

and

$$F = \frac{\left\{1 + \frac{E_0}{1022}\right\}}{\left\{1 + \left(\frac{E_0}{511}\right)^2\right\}}, \quad (3)$$

and

$$E_M = 7.6Z^{0.36}. \quad (4)$$

In the above equations, E_0 is the microscope accelerating voltage, 300 kV in this case, and Z is the atomic number, assumed to be 14 (pure Si) in this case. By plotting Equation 1 as a function of collection angle β , a comparison can be made with the relative thickness values obtained directly on and off the [011] zone axis. Figure 6 is a graph of t/λ as a function of β for the thickness values obtained from the thicker part of the foil (values at the top of each thickness map in Figure 5). In Equation 1, the thickness t acts as a scalar, simply raising or lowering the curve. It can be seen that regardless of the curve height, the thickness values obtained off the zone axis (triangles) fit the form of the curve better than those obtained on the zone axis (circles). The thickness values obtained directly on the [011] zone axis are approximately linear with a slight positive slope. These indicate that erroneous values may result from Equations 1-4 when a sample is oriented directly on a zone axis with strong diffraction contrast.

A set of jump-ratio images acquired from the boxed area in Figure 3, oriented both directly on and off the [011] zone axis using several different objective aperture sizes, is shown in Figure 7. The residual Bragg contrast is severe in the jump-ratio images acquired directly on the [011] zone axis with $\beta = 3$ mrad, moderate when $\beta = 8$ mrad, and is barely visible when $\beta = 16$ mrad. In contrast, the jump-ratio images acquired off the [011] zone axis, but tilted so that the (001) interfaces are still parallel to the electron beam, have no visible residual diffraction contrast and display much better compositional contrast for direct visual interpretation. Interestingly, the thin pure Si layers appear much sharper in all EFI acquired off [011] compared to EFI acquired directly on the [011] zone axis. The box in the upper-

left image represents where a line profile was taken from each image parallel to the long axis of the box (see Figure 9).

Figure 8 is a set of elemental maps acquired under the same conditions as the jump-ratio images shown in Figure 7 using a standard power law routine [18] for background removal. Again, the maps acquired directly on the zone axis with $\beta = 3$ and 8 mrad contain substantial residual diffraction contrast. Only when $\beta = 16$ mrad does the elemental map show enough suppression of residual diffraction contrast to be directly interpretable. This is because the initial image (i.e., the unfiltered or zero-loss image) displayed less Bragg contrast since, as a larger collection angle was employed, more dark-field images were superimposed upon the bright-field image, thereby canceling the diffraction contrast. The elemental maps acquired off the [011] zone axis are again of much better quality for compositional interpretation. The box in the upper-left image represents where a line profile was taken from each image as before (see Figure 9).

Line profiles integrated along the boxes in Figures 7 and 8, parallel to the layers, are shown in Figure 9. Each pixel within the line profile (as well as the digital images) represents 2.6 nm. Inspection of the jump-ratio image line-profiles on and off the zone axis shows that they are comparable only when $\beta = 8$ and 16 mrad. The image acquired directly on the [011] zone axis with $\beta = 3$ mrad is severely affected by residual Bragg contrast and relays little compositional information. Interestingly, the average jump ratio of the line profiles integrated from the images acquired off the [011] zone axis increases with a reduction of collection angle, as seen before [15]. The average jump ratio also increases when directly

on the [011] zone axis, except when $\beta = 3$ mrad. The elemental map line profiles are similar in that the off [011] profiles relay more usable information and are much less affected by residual Bragg contrast than those obtained on the zone axis.

There are three peaks in the line profiles (most easily seen in the off [011] jump-ratio profiles) corresponding to the thin layers of pure Si (marked A, B, and C in the top-right profile in Figure 9). Both A and B are of equal height in the jump-ratio images acquired off [011], but C is lower. The reason that each Si peak is not of equal height is not known, but is probably due to variations in composition. It does not seem likely that the variation is due to imaging conditions since peak C is smaller in all of the line profiles where it can be resolved. The plateaus between the pure Si peaks are the SiGe(X%) layers. Each layer should have a difference of 5% Ge, but the layers do not step down uniformly. The reason for this is again not known, but may be due to **inexact alloying** of Ge with Si, so that the composition change was not exactly 5% between successive layers. We did not attempt to perform independent quantitative measurements of these compositional changes since these are not directly relevant to the main point of this paper.

There are several additional important points to notice from the line profiles in Figure 9. First, the spatial resolution of the Si peaks (A, B, and C) is almost always better for both the jump-ratio images and the elemental maps acquired off (with the interfaces parallel to the beam), rather than directly on the [011] zone axis. Second, the jump-ratio images and elemental maps acquired directly on the zone axis were often severely altered by residual diffraction contrast, except in the case when a large objective aperture (16 mrad) was

employed. This demonstrates that when acquiring EFI directly on a zone axis, a large objective aperture will probably yield the best results for suppression of residual diffraction contrast and, correspondingly, enhancement of compositional contrast. Third, the elemental maps are more **severely** effected by residual Bragg contrast, as previously observed [9-12,15]. The elemental maps acquired directly on the [011] zone axis are affected by residual diffraction contrast for all objective aperture sizes (much more so than the jump-ratio images). Additionally, there is a small trough slightly adjacent to the pure Si peaks in the elemental maps acquired off [011], which diminishes in depth as a larger objective aperture is employed.

The choice of objective aperture size is an important variable in the EFI process since diffraction contrast is often preserved and, when strongly present, does not disappear with increasing energy loss [14]. Therefore, it is important to suppress diffraction contrast in the area to be analyzed as much as possible before attempting to acquire EFI. Additionally, it is important to understand the difference a change in collection angle makes on both EEL spectra and EFI. Table 1 summarizes some of the most important variables affected by a reduction of collection angle when acquiring EEL spectra, jump-ratio images and elemental maps:

- i) ionization intensity – the intensity associated with a core-loss ionization event, i.e., the area beneath an ionization edge,
- ii) signal-to-background ratio (SBR) – the height (area) of an ionization edge in relation to the background intensity,

- iii) signal-to-noise ratio (SNR) [18] – the average intensity value (for a set of pixels in an EFI) divided by the standard deviation (from the same set of pixels),
- iv) residual diffraction contrast – diffraction contrast that is preserved from a bright-field image to raw EFI, jump-ratio images or elemental maps, and
- v) single scattering events – the approximate percentage of electrons which contribute to either a spectrum or image that have suffered only one (elastic or inelastic) scattering event.

As the collection angle is decreased, the ionization intensity decreases in an EEL spectrum (and therefore in an elemental map) simply because there are fewer electrons being collected. However, the SBR in an EEL spectrum increases because as the ionization intensity decreases, the background intensity also decreases [3-5,18,19], which in turn, increases the SBR. This increase in the SBR associated with a reduction of the collection angle increases the jump ratio value (as seen in the off-axis jump-ratio line profiles in Figure 9). The SNR usually decreases in jump-ratio maps as the collection angle is reduced [15], however, it often increases in elemental maps [15,18]. The increase in SNR in elemental maps associated with a reduction of β reverses at some point when the objective aperture becomes exceedingly small [18]. Residual diffraction contrast increases in both jump-ratio images and elemental maps as a smaller collection angle is employed, since more high-angle inelastic scattering (i.e., elastic-inelastic and inelastic-elastic or, otherwise stated, inelastic dark-field images) is removed [14]. Finally, because a reduction of collection angle removes more inelastic-elastic and elastic-inelastic events from EFI, the percentage of single

scattering events in raw EFI, jump-ratio images and elemental maps increases (however, there is still plural scattering when only the 000 beam is collected since inelastic-inelastic scattering events occur).

As an example of the above variables, assume that an objective aperture that isolates the 000 beam is used to acquire EFI. There are both positive and negative attributes to the acquisition of EFI with the collection of only the transmitted beam. On the positive side, the small objective aperture:

- i) isolates more single-scattering events, which are more reliable for quantification,
- ii) normally increases the SNR in elemental maps [15,18],
- iii) increases the SBR, and
- iv) allows for less of an effect of defocus on EFI caused by a change of high tension of the microscope (for a post-column filter).

On the negative side, the small objective aperture:

- i) decreases the SNR in jump-ratio images [15] ,
- ii) increases residual Bragg contrast in EFI [1-14], especially in elemental maps, and
- iii) reduces the ionization edge intensity.

Accounting for the above variables, it seems that the use of an objective aperture with a collection angle of about 8-10 mrad represents a good **compromise** for many imaging situations.

It can be shown that the tilting procedure demonstrated above is completely general and can be used to orient any planar interface to reduce Bragg contrast. Figure 10 is a drawing of a

face-centered cubic (FCC) Kikuchi map with four poles (zone axes) shown. In the lower-right corner the [011] pole is shown with two diffraction patterns tilted off the zone axis. The diffraction pattern above the pole is similar to the experiment shown in this paper where an (001) interface was oriented parallel to the electron beam, but the zone axis was tilted with respect to the optical axis. By keeping the ± 400 reflections symmetric about the optic axis, the planar (001) interface remains parallel to the beam. Similarly, the crystal can be tilted so a (111) planar interface remains parallel to the beam, as illustrated by the diffraction pattern to the lower-left side of the [011] pole. Here the $\pm 2\bar{2}2$ reflections would be made symmetric along the $\pm 1\bar{1}1$ Kikuchi band. As a final example, this titling operation is shown off the [001] pole keeping an (010) planar interface parallel to the electron beam. This is demonstrated in the upper-right corner of Figure 10. Here the ± 040 reflections are symmetric along the ± 020 Kikuchi band.

Finally, it should be highlighted that Hofer and Warbichler [20] have found the effects of residual diffraction contrast can be almost entirely alleviated in jump-ratio images by recording both the pre-edge and post-edge images under rocking beam illumination. This procedure may be used as an alternate method to the above titling procedure when examining a planar interface, however, it may not be advantageous at high magnification since, if the beam is rocked several tens of milliradians perpendicular to the interface, it could result in blurring of the interface. The rocking beam method is certainly an advantageous approach for complex interfaces that are curved and/or tilted in relation to the electron beam.

Conclusions

These experiments demonstrate that it is possible to orient samples so that a planar interface is parallel to the electron beam (optic axis) yet tilted with respect to a zone axis, and that in doing so, diffraction contrast is greatly reduced in both bright-field and energy-filtered TEM images. Importantly, this orientation, in addition to producing substantially reduced diffraction (Bragg) contrast, does not reduce the spatial resolution obtained across a planar interface. The reduced Bragg contrast obtained in bright-field images (with this orientation) is carried over to both jump-ratio images and elemental maps, leaving EFI with nearly no residual diffraction effects. The EFI acquired from a tilted sample provided maximum compositional contrast, which allowed for easier direct interpretation of images and, in most cases, better spatial resolution than in EFI acquired in a zone axis orientation.

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Figure 1. An Al / SIMOX Si (001) structure that is oriented slightly off a [011] zone axis so that diffraction contrast is minimized, but the (001) interfaces are still parallel to the electron beam. (a) Bright-field image (b) corresponding electron diffraction pattern.

Figure 2. A portion of an EEL spectrum showing the Si L_{2,3} edge at 99 eV energy loss. The pre-edge 1, pre-edge 2 and post-edge windows used for EFI acquisition are indicated.

Figure 3. Two bright-field TEM images and diffraction patterns showing the SiGe heterostructure used in this experiment directly on the [011] zone axis and tilted slightly off the zone axis so that the ± 400 reflections are symmetric about the optic axis. The boxes in both images indicate where the diffraction patterns were obtained and where all subsequent EFI were acquired. The white circles superimposed on the lower-left diffraction pattern show the different objective aperture sizes used in this experiment.

Figure 4. Zero-loss images ($\Delta E=10\text{eV}$) acquired directly on the [011] zone axis and tilted off the zone axis so that the (001) interfaces are still parallel to the electron beam. Each image was acquired from the boxes shown in Figure 3 using a collection angle $\beta = 3$ mrad.

Figure 5. A series of thickness maps acquired directly on the [011] zone axis and tilted off the zone axis so that the (001) interfaces are still parallel to the electron beam. Three different objective apertures were used: $\beta = 3, 8$ and 16 mrad.

Figure 6. A graph of t/λ as a function of collection angle β . Notice that the thickness map values acquired off the zone axis (triangles) fit the curve for $t = 85$ nm better than the thickness map values acquired directly on the zone axis (circles).

Figure 7. A series of jump-ratio images acquired directly on the [011] zone axis and tilted slightly off the zone axis so that the (001) interfaces are still parallel to the electron beam. Three separate objective apertures were used: $\beta = 3, 8$ and 16 mrad. The box in the upper-left image shows where the line profiles in Figure 9 were taken.

Figure 8. A series of elemental maps acquired directly on the [011] zone axis and tilted off the zone axis so that the (001) interfaces are still parallel to the electron beam. Three separate objective apertures were used: $\beta = 3, 8$ and 16 mrad. The box in the upper-left hand image shows where the line profiles in Figure 9 were taken.

Figure 9. Line profiles from the areas enclosed in the boxes in Figures 7 and 8. Notice that the spatial resolution of the peaks (due to the small layers of pure Si) is generally higher for the jump-ratio images and elemental maps acquired slightly off the [011] zone axis than for the images acquired directly on the zone axis.

Figure 10. A schematic diagram of an face-centered cubic (FCC) Kikuchi map illustrating the tilting procedure for orienting a planar interface so that it is parallel to the electron beam, but not directly on a zone axis (after Figure 19.5 in [21]).

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Table 1. Changes incurred in signal(intensity) and contrast in EEL spectra, jump-ratio images and elemental maps as the objective aperture collection angle β is decreased. The symbol \uparrow means increase and \downarrow means decrease.

REDUCTION OF β	Ionization intensity	Signal-to- background ratio (SBR)	Signal-to- noise ratio (SNR)	Residual diffraction contrast	Single scattering events
EEL spectrum	\downarrow	\uparrow	\uparrow or \downarrow	-	\uparrow
Jump-ratio image	\downarrow	\uparrow	\downarrow	\uparrow	\uparrow
Elemental map	\downarrow	\uparrow	\uparrow or \downarrow	$\uparrow\uparrow$	\uparrow