Simultaneous orientation and thickness mapping in transmission electron microscopy

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In this paper we introduce an approach for simultaneous thickness and orientation mapping of crystalline samples by means of transmission electron microscopy. We show that local thickness and orientation values can be extracted from experimental dark-field (DF) image data acquired at different specimen tilts. The method has been implemented to automatically acquire the necessary data and then map thickness and crystal orientation for a given region of interest. We have applied this technique to a specimen prepared from a commercial semiconductor device, containing multiple 22 nm technology transistor structures. The performance and limitations of our method are discussed and compared to those of other techniques available.

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1. Introduction

Transmission electron microscopy (TEM) is a powerful tool for investigating the atomic structure and morphology of nano- and micro-objects. Specimen prepared for TEM should be reasonably thin in order to be transparent for the electron beam. The thickness of the sample is an important parameter one should account for during the experiment and for quantitative data analysis. Potential mapping by off-axis holography, elemental mapping by energy-filtered TEM, or the calibration of inelastic mean free path lengths all rely on the ability to accurately map the specimen thicknesses. For a quantitative analysis of high-resolution TEM images, e.g. by comparison with multislice simulations, it is important to know both the specimen thickness and also the precise orientation of the sample. There are several techniques available to estimate the specimen thickness by TEM [1]. These methods can be divided into several categories, depending on whether they rely on energy-filtered images [2], convergent beam electron diffraction (CBED) [3–5], electron energy-loss spectroscopy (EELS) data [6,7], or energy-dispersive X-ray spectroscopy (EDXS) data [8,9]. None of these techniques is universally applicable. CBED-based methods, for example, may analyse the crystal thickness at individual points, but deliver reliable thickness estimates only if the specimen is rather thick (typically more than 50 nm) and the unit cell small (to avoid overlapping CBED discs). The most popular technique for measuring specimen thickness is based on EELS [10]. On microscopes equipped with an energy filter this method is easy to implement both experimentally and computationally but it may have an error of up to ±20%, depending also on how accurately the inelastic mean free path is known. One of the reasons for this inaccuracy is that thin crystalline samples may bend due to lattice relaxation. Bend contours which are a consequence of variations in the local lattice orientation, can strongly influence the thickness values obtained by EELS.

Such bend contours appear because elastic scattering of electrons in crystalline materials is very sensitive to the local lattice orientation. Variations in the local lattice orientation can be visualized by orientation maps. Routine crystal orientation mapping on a relatively coarse scale was first realized in the TEM by Dingley and Wright [11–13] and has initially been commercialized by TexSEM Laboratories (TSL) as Automated Crystallography for TEM (ACT). This method uses a set of DF images acquired for many different illumination tilt angles and constructs a set of synthetic diffraction patterns from it which it then analyses in terms of absolute lattice orientation. Since then many other TEM-based orientation mapping techniques have been developed. Most of these techniques rely on the analysis of maps of either individually recorded or synthetic diffraction patterns by either a geometric interpretation of spot positions, or by matching each pattern to a large library of pre-generated template patterns [14]. Recording individual diffraction patterns can be done with parallel (nano-beam diffraction (NBD) [14]) or convergent (CBED [15]) illumination, or by combining one of these setups with precession of the
illumination tilt angle [14]. Recently, also a three-dimensional (3D) grain orientation mapping technique based on this approach has been developed [16], where the orientations of individual voxels (volume elements) was reconstructed from more than 100,000, i.e. \(10^5\) DF images, acquired for many different directions of the incident illumination and sample tilts. It has also been shown that the orientation analysis becomes more precise when combining the spot pattern analysis with the analysis of the Kikuchi pattern that appears in its background [17].

In this paper we report a technique which simultaneously delivers maps of both specimen thickness and crystal orientation. In contrast to orientation mapping in polycrystalline materials, our approach only maps local deviations from a given reference crystal lattice orientation. In most experiments related to the semiconductor industry the orientation of the specimen is fixed, but in order to quantify the strain state of the specimen it is important to

![Image of DF tilt series used for analysis](image)

**Fig. 1.** Top: one of the images from the DF tilt series used for this analysis. The image with the highest mean intensity has been selected. Middle: rocking curves extracted from the data at the three positions indicated in the image above and plotted as a function of tilt angle. Bottom: power spectra calibrated in nm corresponding to the three spectra shown above. The abscissa (z) in these graphs thus reflects the real space wave number describing the reciprocal space oscillations in the rocking curves. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this article.)
measure local changes in orientation. With the approach presented below such local orientation changes can be mapped with a precision of <0.1°.

Very similar to CBED-based thickness mapping, our approach is based on the analysis of rocking curve information. However, in contrast to CBED, where one CCD exposure acquires a 2D rocking curve (of very limited tilt range) for each of the diffracted beams at a single point on the specimen, we propose to extract one-dimensional rocking curves from dark-field (DF) images acquired at slightly different specimen tilts, covering a tilt range of only a few degrees. A major advantage of our approach over the analysis of CBED patterns is that rocking curve information from very many positions on the specimen is collected in parallel. Another advantage is that the tilt range over which the rocking curve information may be collected is not limited by the distance between neighboring reflections, which allows the thickness and orientation of much thinner samples to be determined. In case of a bent specimen a thickness measurement by 2-beam CBED rocking curve analysis requires the sample to be reoriented when moving to a different position on the sample, which often also leads to a movement of the probe on the specimen. The technique proposed here acquires a tilt series in image mode. Any specimen shifts during tilting can thus be compensated in a very straightforward manner.

In Section 2, we discuss in some more detail the sample preparation and data acquisition, and also the concept and different tools used for data analysis. In Section 3, we present the recovered thickness and orientation maps and discuss possible applications in Section 4.

2. Method

2.1. Experiment

The sample from which the experimental data was collected was prepared from an off-the-shelf Central Processing Unit (CPU), containing 22-nm node metal-oxide semiconductor field-effect transistor structures (MOSFETs). The Si substrate of the microchip was cut parallel to its edges ([110] crystallographic planes) into millimeter sized pieces by using a diamond saw. This aligned the cutting direction conjugate to the orientation of the Si source-drain fins. A detailed description of the different components of such MOSFET structures can be found in the literature [18–20]. The diagram showing different parts of MOSFET structure is shown as inset in Fig. 1. A thin wedge-shaped sample was prepared by means of automated tripod polishing using the MultiPrep™ system (Allied High Tech Products, Inc.), followed by ion milling using a PIPS Model 691 (Gatan Inc., Pleasanton, CA, USA) at low ion beam energy. The polishing angle was set to approximately 1.5°. The thickness at the thin area of the wedge was controlled via means of automatized tripod polishing using the MultiPrep system (Allied High Tech Products, Inc.), followed by ion milling using a PIPS Model 691 (Gatan Inc., Pleasanton, CA, USA) at low ion beam energy. The polishing angle was set to approximately 1.5°. The range of scattering angles passing through this objective aperture corresponded approximately to the size of the first Bril- louin zone. The in-column energy filter was used to exclude most inelastically scattered electrons from contributing to the images. The images comprising the tilt-series were subsequently aligned with respect to each other by using the Statistically Determined Spatial Drift Correction (SDSR) routine [24], in order to remove any sample drift. This routine proved to be much more robust when compared to standard cross-correlation between adjacent images for the case of this DF tilt series. Fine alignment was achieved by using the FRWR tools (Full Resolution Wave Reconstruction) plug-in [25] which allowed manual tracking and compensating of the displacement between adjacent images in the data stack.

2.2. Data analysis

2.2.1. Calculation of thickness map

Since 2-beam rocking curves are usually oscillatory in nature (see Fig. 1) we expect to see well-defined peaks in the power spectrum of such a rocking curve, i.e. the modulus of its Fourier transform. The power spectra of rocking curves at different sample positions are shown at the bottom of Fig. 1 as blue dots. The fits to the power spectra (according to expression (2)) are displayed as solid green lines. Fig. 2 illustrates how a dark-field tilt series samples reciprocal space. The vertical reciprocal space coordinate k_z at which the Ewald sphere intersects the relrod of reflection \( \mathbf{g} \) is related to the holder tilt angle \( \alpha \) according to

\[
k_z = |\mathbf{g}| \sin(\alpha) \sin(\phi)
\]

where \( \phi \) is the angle between the vector \( \mathbf{g} \) with the holder axis. The oscillatory behavior of the extracted rocking curve is largely due to the oscillations in the relrod (see Fig. 2). While in kinematic and 2-beam dynamical diffraction theory, the oscillations in the rocking curve are directly related to the amplitude variations along the relrod, in many-beam dynamical theory the rocking curve becomes much more complicated. In order to determine the specimen thickness that gives rise to the oscillations in a given rocking curve \( I(k_z) \), we fitted an analytical function \( f(z) \) comprising the sum of 5 Gaussians to \( y(z) \), the power spectrum of \( I(k_z) \):
In order to reduce the effect of noise in the rocking curve, the data was slightly blurred after computing the power spectra of all rocking curves.

2.2.2. Calibration of thickness map

To calibrate the thickness map i.e. to determine the specimen thickness that a certain value of \( b \) in our fitting model (2) corresponds to, we applied the fitting procedure described above to rocking curves simulated for known specimen thicknesses. For simulating the rocking curves we have applied simple 2-beam theory, for which the intensity of the diffracted beam is given by [26]

\[
I^2_{\text{2-beam}}(\alpha) = I_0(\lambda U_k)^2 \frac{\sin^2(\alpha \sqrt{\frac{1}{2} (sg(\alpha)^2 + 2^2 b^2 U_k^2)})}{sg(\alpha)^2 + 2^2 b^2 U_k^2} \quad (3)
\]

where \( I_0 \) denotes an overall intensity scale that depends on exposure time and incident electron beam current density, \( \lambda \) is the wavelength of the fast electrons (\( \lambda \) (120 kV) = 3.35 pm with relativistic effects taken into account), \( U_k \) is the structure factor of the scattering material (silicon) that corresponds to the reciprocal vector \( \vec{g} \) in the diffraction plane, \( t \) is the local thickness of the specimen, and \( sg \) denotes the effective excitation error which can be calculated in the following way:

\[
sg(\alpha) = - \frac{\lambda}{2} (\vec{g} \cdot \vec{k}_{\alpha}) \quad (4)
\]

Here \( \vec{k}_{\alpha} \) denotes the transverse component of the incident wave vector, i.e. the \( \{k_x, k_y\} \) component of the wave vector that lies in the plane shown in Fig. 2. The argument of the sine function in expression (3) shows that the minima of the intensity occur when \( t \sqrt{\frac{1}{2}} \) \( \frac{\lambda}{U_k^2} (\text{an integer number}) \) this behavior is also frequently used for fitting specimen thickness from 2-beam CBED data.

In the reference frame of the tilted crystal the vector \( \vec{k} \) is given by

\[
\vec{k}(\alpha) = \frac{\sin(\alpha - \alpha_0)}{\lambda} \hat{h}_2 \quad (5)
\]

In Expression (5) \( \hat{h}_2 \) is a horizontal unit vector defined in the reference frame of the crystal that is perpendicular to the axis of the specimen holder, i.e. \( \hat{h}_2 \) is normal to both the holder axis and the nominal zone axis of the crystal. This also means that for reflections \( \vec{g} \) that lie parallel to the direction of the holder axis the excitation error \( sg(\alpha) \) will remain constant while the holder is tilted, since in that case \( \vec{g} \cdot \hat{h}_2 = 0 \). In the above expression the argument \( \alpha \) is again the tilt angle of the sample holder, and \( \alpha_0 \) determines the component of the local deviation of the specimen orientation that is perpendicular to the holder axis. When using only one reflection we can only determine deviations in the specimen orientation that correspond to rotations about the holder axis. Measuring the component of the specimen tilt along the holder axis would require a DF tilt series for a second reflection.

As can be seen from Fig. 3, for specimen thicknesses between 30 and 100 nm the calibration factor \( r=\lambda/b \) has an almost constant value of \( r \approx 16.5 \) (\( t = rb \)) For thicknesses outside this range, this

\[
\begin{array}{cccccc}
\text{y-power spectra} & a_1 & a_2 & a_3 & b \text{ (nm)} & c \text{ (nm)} \\
\text{Start point} & \text{max(y)} & \text{max(y)} & \text{max(y)} & 49.5 & 16.5 \\
\end{array}
\]

The above model takes into account that the power spectrum \( y(z) \) is symmetrical with respect to the origin, i.e. the position of zero frequency. Since \( k_z \) is given in units of nm \(^{-1} \), the coordinate of the power spectrum of the rocking curve, \( z \), is given in units of nm. The parameter \( b \) is expected to be approximately proportional to the specimen thickness. Thick regions correspond to large values of \( b \) and low thicknesses to small values of \( b \).

The fitting was done in MATLAB™ (The Mathworks, Inc.) making use of the Curve Fitting Toolbox. For fitting the nonlinear least squares method and the trust region algorithms were used. The standard deviations \( c \) and the amplitudes \( a \) of the Gaussians were constrained to be positive. The starting values for these parameters are described in the following table:

\[
\begin{array}{cccccc}
\text{y-power spectra} & a_1 & a_2 & a_3 & b \text{ (nm)} & c \text{ (nm)} \\
\text{Start point} & \text{max(y)} & \text{max(y)} & \text{max(y)} & 49.5 & 16.5 \\
\end{array}
\]
The calibration factor is different and must be read from the plot shown in Fig. 3.

In our numerical data analysis we have decided to use expression (2) rather than expression (3) because it has proven to be much more robust against deviations from a perfect 2-beam diffraction condition. While the sinc2-like function used in expression (3) to describe the 2-beam rocking curve \( I(\alpha_f) \) fixes the heights of the side lobes in relation to that of the central peak, expression (2) is much more flexible, and can more easily account for deviations from a perfect 2-beam condition, or the potential presence of a background in the rocking curve data \( I(\alpha) \), due to, for example, thermal diffuse scattering.

Applying the calibration data shown in Fig. 3 we are now able to quantify the specimen thickness on an absolute scale. Fig. 4 shows the calibrated thickness map reconstructed from the DF tilt series. One can see that the thickness increases gradually from the edge into the sample, verifying that the sample is indeed wedge-shaped, as we would expect. One should also note that, as with any other thickness measuring technique, the thickness estimated this way represents the projected thickness of a potentially tilted slab. Measured normal to the specimen surface the actual thickness of the crystal may differ from the values shown here. However, as described above, in the current experiment the sample was cut in such a way that the nominal specimen surface normal is parallel to the \([110] \) zone axis, so that such projection effects can largely be neglected.

To verify that the final thickness map truly corresponds to the best possible solution we carried out a multiple local minima analysis. The cost-function we are trying to minimize by calculating the best fit can show several local minima. To determine the parameters corresponding to all of these minima by an extensive multidimensional search at each pixel in the data stack is a computationally very expensive procedure and is therefore considered inefficient. However, we did check our results using the MATLAB function MultiStart [27] from the Global Optimization Toolbox. MultiStart runs the local solver for many randomly generated sets of starting points, sampling as many possible basins of attraction as possible. The thickness map presented earlier did indeed correspond to the best solutions found by this global search scheme. We also estimated the thickness of the sample by conventional EELS-based \( t/\lambda \) mapping. Values calculated by both methods are in good agreement with each other.

2.2.3. Calculation of orientation map

In order to measure changes in the local crystal orientation we decided to fit a Gaussian functions to each of the rocking curves. As can be seen from Fig. 1, for thin areas the intensities of the side lobes in the rocking curves are quite weak, and the signal is dominated by the central peak at the exact Bragg condition. At higher thicknesses the side lobes become stronger and move closer to the central peak but the rocking curves remain largely symmetrical. This makes fitting the angle that corresponds to the exact Bragg condition quite accurate. Deviations in the position of the center of the Gaussian curve translate directly to changes in

![Fig. 3. Plot of the calibration factor \( r=t/b \) for different specimen thicknesses. For each thickness \( t \) a rocking curve was simulated using expression (3) and the parameter \( b \) was fitted to its power spectrum using expression (2).](image-url)

![Fig. 4. Figures from top to bottom: (a) thickness map (in Å), (b) orientation map (in °), (c) and orientation map profiles. (a) and (b) In places where the highest intensity of the rocking curve was below a threshold of 40 counts, the thickness and orientation were set to zero in order to enhance the visibility of the Si fins, and because thickness and orientation outside the crystalline area are meaningless, of course. (c) The orientation map profiles extracted from individual Si fins were averaged over a width of 10 nm. The color scheme of the plots corresponds to the colored dots shown in (b).](image-url)
the local crystal orientation \( \alpha_0 \) in expression (5).

The precision of the orientation map is expected to be <1 mrad, because it depends not directly on the reproducibility of a specific goniometer tilt (which is typically about 0.1°), but more on the statistical fluctuation of the tilt accuracy over those parts of the tilt series for which the intensity of the DF images is high, because those intensities will then contribute significantly to the Gaussian fit. The accuracy of the orientation determination depends on the accuracy of the calibration of the goniometer tilt and the calibration of the beam tilt. Both should be <1 mrad.

For processing the DF images as described above an interactive application featuring a graphical user interface has been written in MATLAB. In this application the image data can be imported in MATLAB’s workspace and the sample area for which the thickness and orientation map is desired can be selected. To read digital electron micrographs in DM3 format, the code from Sigworth was used [28]. To view DM3 data stacks slice by slice in MATLAB a small viewer was developed.

3. Results and discussion

Although applied here only to Si(110) we want point out that this method may work equally well for different single crystalline materials (or within a single grain of a polycrystalline material) even if these are of different space groups. The only requirement is that for the experiment a reflection is chosen which is not collinear with the holder tilt axis. This method is, in principle, as widely applicable as thickness measurement by CBED, except that it provides a map of both thickness and orientation over a large field of view, instead of just a single measurement. The field of view is either limited by the detector, or by the size of the single crystalline region being investigated.

However, in contrast to thickness measurement by CBED where the camera length can be chosen large enough to sample even very fine fringes within the CBED disc the method presented here may not be able to reliably determine the thickness in case the rocking curve is not sampled finely enough. As one can see in Fig. 2, at large specimen thicknesses the distance between minima in the rocking curve may be of the order or even smaller than the tilt steps. In that case the intensity variations which enable us to determine the local thickness may not be detectable. Well-controlled finer tilt steps, or a restriction to specimen thicknesses and diffraction conditions where the 2-beam thickness oscillations can still be sampled by the goniometer tilt, could solve this problem.

The excitation of other beams (e.g. higher order Laue zone (HOLZ) reflections) may introduce sharp features in the rocking curves, especially in thick specimen areas. At lower thickness HOLZ lines are not quite as sharp. For silicon and the particular set of parameters applied in this experiment we estimate that the method should work up to a thicknesses of 120 nm. In order to be able to detect very low thicknesses the tilt range may have to be increased, since the 2-beam rocking curve, which at low thicknesses becomes very similar to the kinematic rocking curve extends to rather large tilt angles.

While orientation mapping using this method is not affected by any of the above-mentioned problems, it is important to note that the range of misorientations that will be detected with this technique is limited by the tilt range for which DF images are collected, at least, if DF images from only a single reflection are being acquired. We also want to point out that the sensitivity of this method to slight changes in orientation increases when using higher order reflections further away from the central beam (but still non-collinear with the holder axis).

The orientation profiles shown in Fig. 4c were integrated in width over 3 pixels and plotted for each Si fin. These orientation profiles show quite interesting behaviors. It seems that some Si fins are bent upward and some downward. The possible explanation of this phenomenon could be deformations of the source-drain structure close to the Si edge. These measurements of an out-of-plane orientation component are complementary to the measurement of in-plane rotation of a similar kind of structure observed by Hytch et al. [29] via dark-field off-axis holoography, although those structures were considerably larger. The structure observed in that work [29] was a special test structure where a contact etch stop layer (CESL) induced strain and some deformation of similarly shaped, but much larger fins. The fins in that structure had a length of approximately 300 nm, whereas they are only 80 nm long and laterally much thinner in the commercial device we have investigated in the present work. From the orientation map one can also see that the orientation varies slightly from left to right. This likely means that the sample is also slightly bent in the horizontal direction. However in the current data set only tilts about the [002]-axis have been measured, and information about any lattice tilt about the [220]-axis is not available. To get the horizontal ([220]-axis-) component of the tilt one needs DF data from another non-collinear reflection. However the sample was bent presumably mostly along the vertical direction, so small deviations along the horizontal directions are negligible. We have also recorded dark-field tilt series for the [002] and [111] reflections. The [111] reflection was not commensurate with the tilt axis. The analysis of that data showed that it was impossible to precisely identify the position of the exact Bragg condition for the [111] reflection. This confirms the obvious fact that for reliable orientation mapping one needs to use reflections for which the product \( g_{\text{bulk}} \cdot h \) is rather large, i.e. the vector should be nearly perpendicular to the tilt axis or refer to a high order reflection (preferably even a HOLZ reflection).

In principle it is possible to repeat the experiment by adjusting the position of the specimen in the holder. However such a procedure would be quite time consuming and possibly not very precise. An alternative approach to perform this experiment for multiple reflections would be based on the usage of a rotation holder which is computer controlled in order to allow full automation.

The remaining fluctuations in the orientation map outside of the leftmost fin could stem from the fact that we have simply used an intensity threshold to distinguish between crystalline and amorphous or polycrystalline material, or crystalline material of very different orientations. While amorphous material may contribute to the intensity in DF images, it will not have the type of rocking curve features we expect from a crystal. Thus only crystalline materials can be analysed by the technique presented here. Thickness mapping by our technique is largely insensitive to the thickness of amorphous layers on the crystal’s surface.

Finally, the thickness and orientation maps obtained by the approach introduced above can be used as a starting guess for a Bloch-wave refinement algorithm recently developed by Pennington et al. [30].

4. Summary

In this paper we described a practical approach for precise and accurate simultaneous thickness and orientation mapping of (single grain) crystalline materials in TEM. By using the tools described in this work it is possible to obtain thickness and orientation maps in an automated way. Since this method is based on the analysis of rocking curve information it is sensitive to the thickness and orientation of the crystalline material only. Any presence of amorphous surface layers would produce a constant
background to the rocking curve and thus does not affect the results, except for possibly decreasing the signal/noise ratio.

The performance was demonstrated on a state-of-the-art semiconducting device. The technique is expected to be very useful towards the characterization of the 3-dimensional strain in crystalline materials, since the orientation information provided by this technique can be used to supplement 2D strain information obtained by other techniques [21,31–33].

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