https://doi.org/10.1017/S1551929512000363 Published online by Cambridge University Press

# High-Resolution Metrology in the TEM

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## Introduction

The transmission electron microscope (TEM) is well known as the technique of choice for visualization and measurement of features at near-atomic length scales, particularly for semiconductor devices. For example, a critical measurement of interest may be the thickness of the gate oxide in a transistor. The accuracy of these measurements is based on calibrated distances at each magnification. The term accuracy conveys the extent to which the measurement minimizes the difference between the measured value and the true value. The associated term precision is the closeness of agreement in a series of measurements locating the end-points of a measurement line [1]. This article describes a method that increases the accuracy of metrology measurements applied to a high-resolution TEM image.

## Methods and Materials

For this article, a TEM sample was prepared from a CPU chip pulled from a computer headed to recycling. The packaging around the chip was dissolved by dipping the chip in hot nitric acid. A portion of the chip was identified as a candidate region for a high density of transistors, and the upper-level metalization and dieletric layers were removed to facilitate the identification of a row of transistors. After identifying a suitable area, an electron transparent cross-sectional TEM specimen was prepared using a dual-beam FIB with *in-situ* lift-out capability. Subsequent TEM imaging was performed using an FEI Tecnai F30 TEM operated at 300 kV in phase contrast mode at Scherzer defocus.

Consider the as-recorded TEM image of a gate oxide structure shown Figure 1 where the substrate Si, amorphous $SiO_2$ dielectric layer, and the doped poly-crystalline Si gate are captured within the field of view. The sample was loaded into the TEM holder such that when tilted to the substrate Si ⟨110⟩ zone axis, the Si [001] direction is oriented nearly "up," as viewed in the image at this magnification. The microscope was aligned and configured for high-quality phase-contrast imaging. A 1k × 1k digital image was recorded using a Gatan MSC 1k × 1k CCD camera with an acquisition time of 0.4 seconds in preparation for post-processing using Digital Micrograph software from the Gatan.

## Calibration

The digital image acquisition software has a nominal calibration for each TEM magnification (145kX, 245kX, 290kX, etc.). For example, Figure 1 was acquired at a nominal magnification of 295kX, which allowed a size bar to be drawn where 10 nm is equal to about 166 pixels. However, for the highest-accuracy measurements, basing the image calibration on a previously calibrated value is not ideal.

A better approach is to use an internal calibration inherent to the image. Fortunately for microscopists in the semiconductor industry, most devices are manufactured along specific crystallographic directions of the substrate, permitting the calibration of the image based on known atomic spacings in the substrate. If the substrate is assumed to have lattice parameters characteristic of the bulk material, and the image is of high enough magnification to resolve the substrate lattice, the lattice itself can be used as the internal calibration.

In principle, the procedure for calibrating an image is easy: pick two points of a known distance apart and use the known distance between the two points to calibrate the equivalent distance in the image at a particular magnification.

## Measurement Precision

The precision of the measurement then becomes the important issue. Consider Figure 2, which is an enlargement of the Si substrate of Figure 1. The image is somewhat pixilated, making a determination of the atomic column center difficult. Furthermore the real and image artifact (aberrated) contrast, apparent in Figure 1, produce variations throughout the image, which makes a representative measurement between rows of atoms difficult. An alternative and potentially more accurate method would be to convert the “real space” image of the substrate into a “reciprocal space” image via a Fourier transform as shown in Figure 3 [2]. Each spot in the Fourier transform, also known

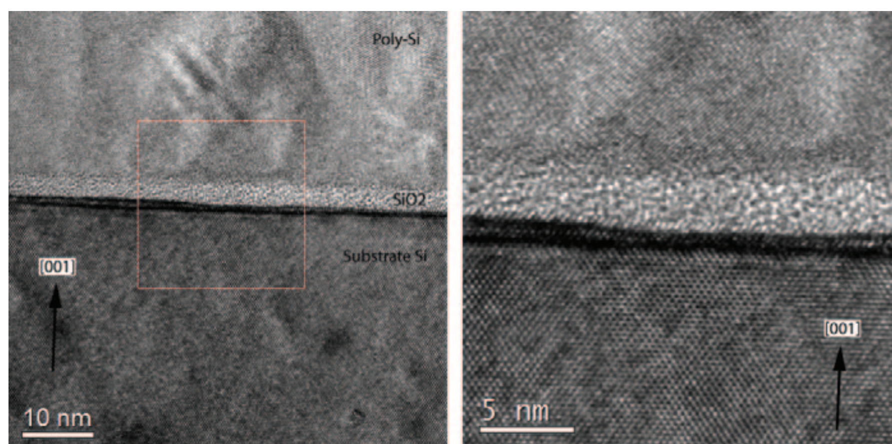


**Figure 1:** As-recorded high-resolution TEM image of a transistor gate (left) showing the substrate Si, the dielectric amorphous $SiO_2$ layer, and the doped polycrystalline Si gate. On the right is an enlarged central region showing the Si lattice of the substrate. The dark line at the top of the substrate is caused by strain giving rise to local differences in electron scattering. The specimen was oriented such that the substrate ⟨110⟩ is along the beam direction. The [001] direction of the substrate is indicated.

 doi:10.1017/S1551929512000363 

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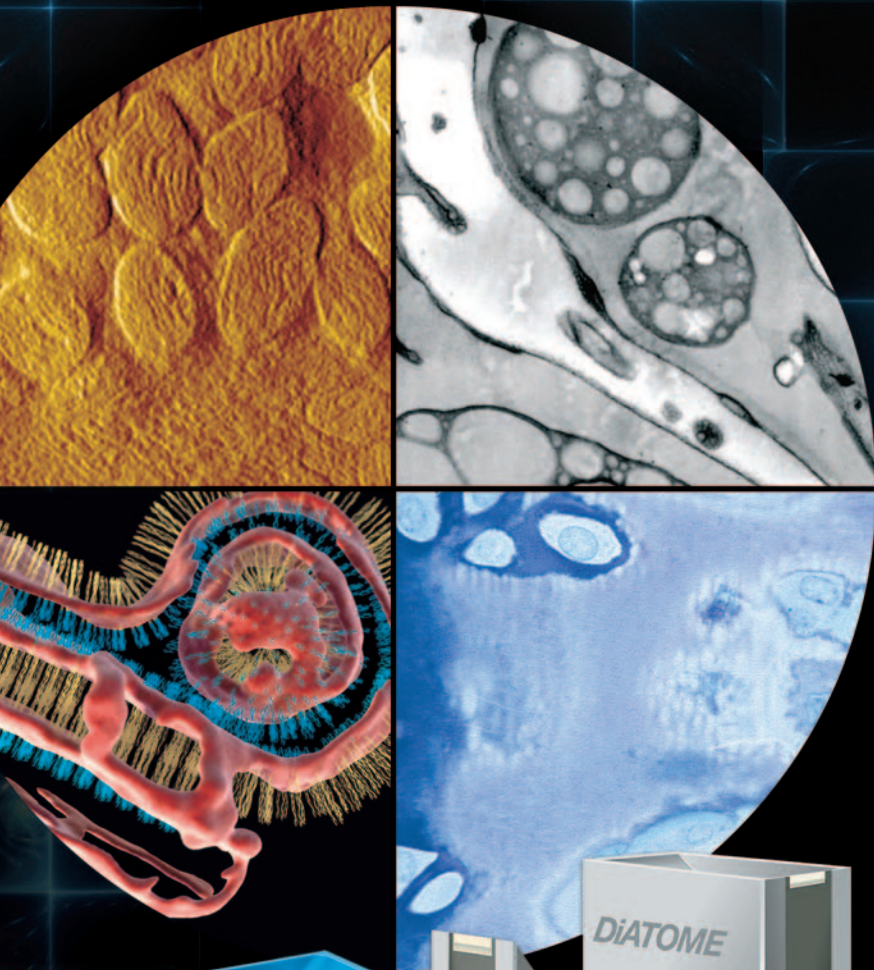

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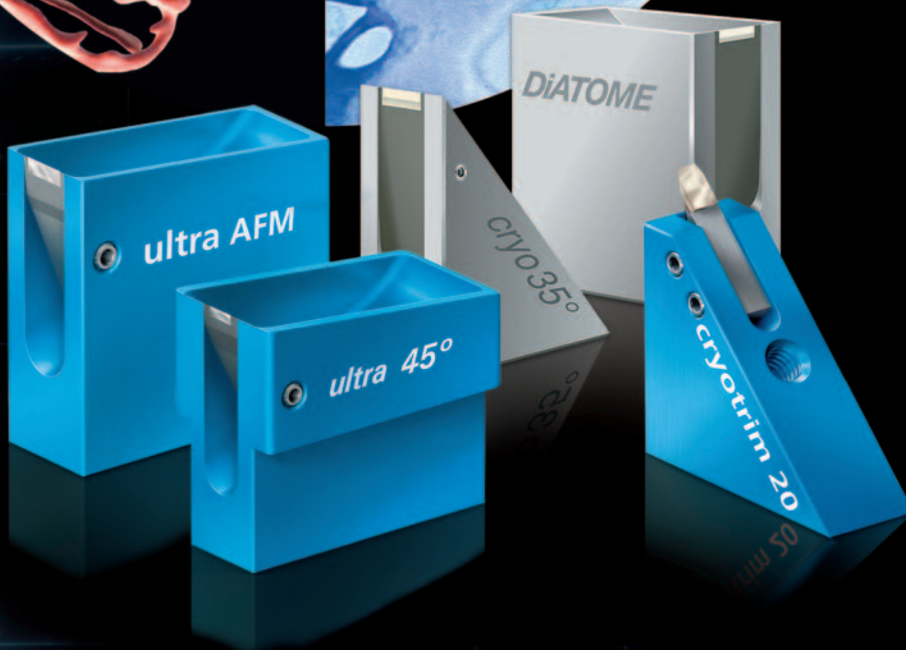


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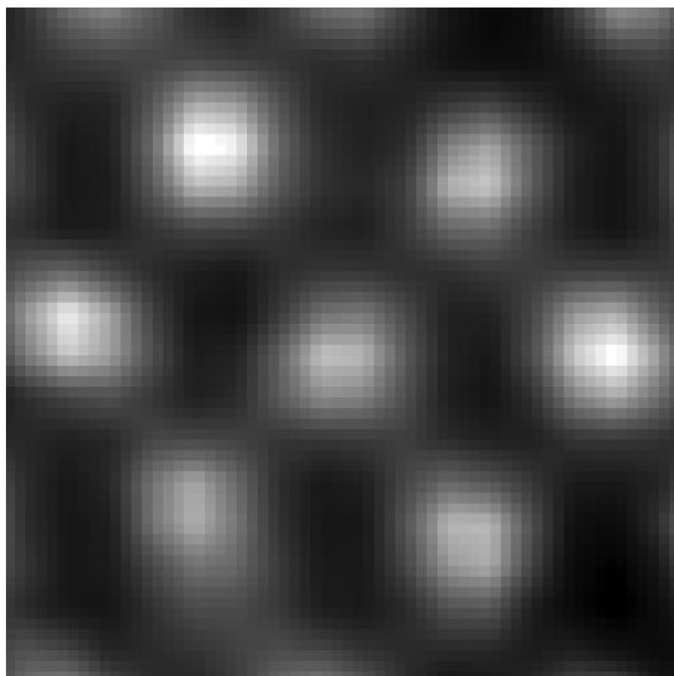

**Figure 2:** Digital enlargement of the substrate imaged along the ⟨110⟩ zone axis in Figure 1. The degree of pixelation and non-uniformity in the atomic column centers make drawing reliable calibration lines difficult. Note that the resolution of the microscope was not sufficient to resolve the dumbbells.

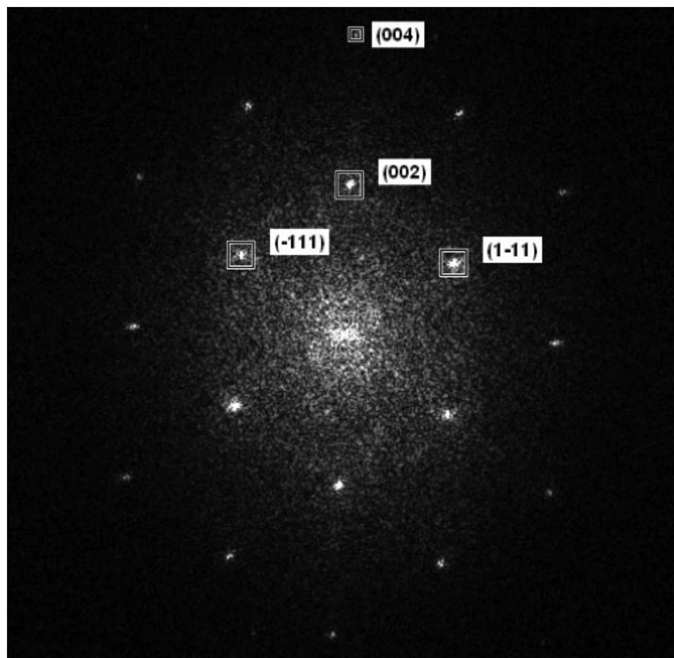


**Figure 3:** Digital diffractogram calculated from the substrate of Figure 1. Pattern shows the symmetry of the Si ⟨110⟩ zone axis pattern. Several spots are indexed and shown with bounding boxes used for the local peak-finding algorithm.

as a digital diffractogram, represents the average periodicity of the corresponding "rows" of atoms in the image. By considering all the periodicies within the selected region of the substrate, rather than just a distance between two points, the random contrast fluctuations and apparent strains in the substrate are averaged out, yielding a more representative spot to measure for the calibration.

The precision in measuring a spot in reciprocal space is still a challenge. One could take the brightest pixel or what appears to be the center pixel of the spot as the location corresponding to the true periodicity of the image, but this is subject to operator error. Rather, the optimum spot location can be determined with sub-pixel accuracy by interpolating a two-dimensional surface in the vicinity of the diffractogram spot from which a maximum intensity location can be identified [3]. This feature is built into the DiffPack plug-in for the Digital Micrograph software package from Gatan. Based on the crystallographic structure of the sample, the reciprocal space image can be indexed in the same way as for a standard selected area diffraction pattern and calibrated based on those known bulk d-spacings. In this case, to calibrate the sample of a Si [001] substrate along its surface normal, the $d_{004}$ spacing is used, and thus for cubic crystals:

$$d_{004}^{Si} = \frac{a_0^{Si}}{\sqrt{h^2 + k^2 + l^2}} = 1.3571 \text{ Å}$$

where $a_0^{Si}$ is the bulk lattice constant of Si, and $h,k,l$ are the Miller indices of the planes of interest. With the FFT calibrated in reciprocal space units (pixels/nm), through the properties of the FFT, the real space image calibration is equal to the inverse of the total calibrated width of the FFT [4].

## Image Rotation

After the raw high-resolution TEM image has been calibrated based on the substrate lattice, the image can be digitally processed to enable more precise measurements of structures contained within the image. To more accurately measure the distances between parallel interfaces, the image can be rotated to make the interfaces parallel to a side of the image. Then measurement lines may be drawn parallel to an edge of the rotated image, removing operator variability in trying to draw lines perpendicular to tilted interfaces. Images of samples from the semi-conductor industry are particularly conducive to this step because the films typically are grown parallel to the substrate surface, which itself is a specific crystallographic plane. In this case, one need only rotate the image according to the angle between an appropriate row of spots in the Fourier transform and the horizontal axis. For example, the angle to rotate Figure 1 would be that between a line drawn from the (–111) spot and the (1–11) spot and the horizontal in Figure 3.

## Metrology

After calibrating the high-resolution image and rotating it so that vertical (or horizontal) features can be used to measure lengths, proper metrology can begin. The simplest technique is to draw a single-pixel-wide line in the calibrated image spanning the feature of interest and read its length with the Digital Micrograph software. This type of measurement is shown in Figure 4 as black lines at two different locations. Alternatively, one can average the intensity values of pixels in rows parallel to the interfaces, as indicated by the box at the left of Figure 4. From these averaged intensities, an intensity profile can be plotted spanning the interfaces, as shown at the bottom of Figure 4. The gate oxide layer thickness can now be measured as the distance (2.58 nm) across the higher intensity (bright) portion of the intensity profile. Note that the double-dip dark lines in the image are seen as local minima near 7 nm and 8 nm. These lines are due to strain within the crystalline substrate and are therefore not part of the critical measurement of the amorphous $SiO_2$ gate-oxide thickness.

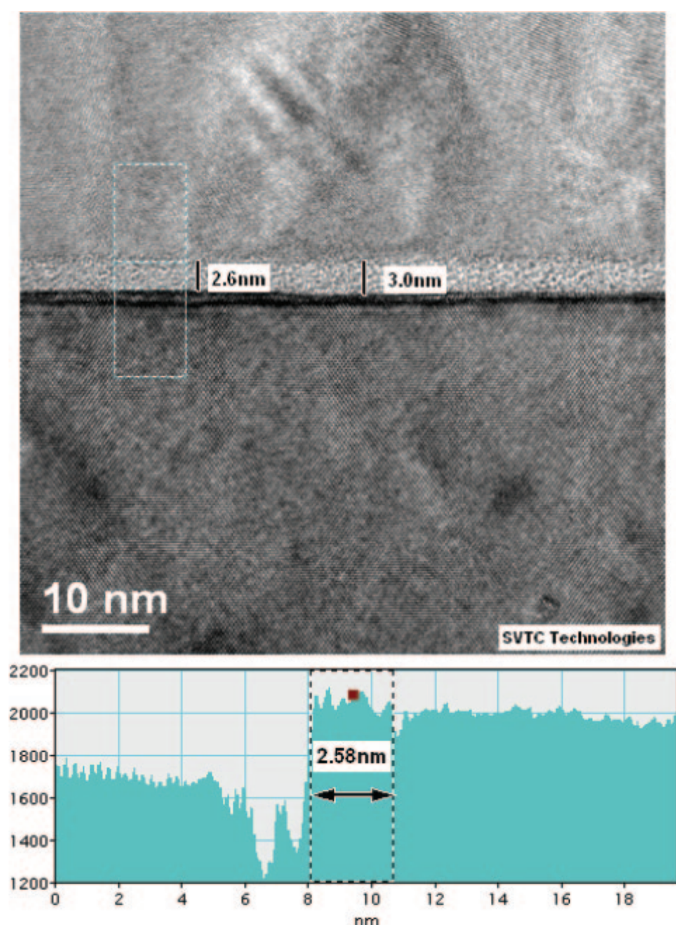


**Figure 4:** Calibrated image has been rotated to ensure substrate [001] direction is up in the image and then cropped to the maximum size without empty regions. Line measurements and extended intensity trace measurement are shown indicating different methods of measuring a distance.

## Discussion

Typical TEM magnification calibration methods have been reported using both real space and reciprocal space methods [5, 6]. Both techniques are viable, but they provide only nominal calibrations applied to each magnification. The use of magnification calibrations acquired separately under nearly identical conditions has been suggested to only be as good as 5% [7, 8] or even 10% [9].

A single crystal substrate tilted to a known orientation may be used as an internal reference and provides the most accurate method of calibrating a high-resolution image. A quantitative measure of the accuracy is difficult to make as it is the difference between the measured value and the known value. However, for the purposes of an estimate, a series of 17 overlapping diffractograms were calculated throughout the crystalline Si substrate of Figure 1 after calibration. The [004] spot was measured in each diffractogram, with the average calculated to be less than 0.2% different than the calibrated value (difference of 0.003 Å). This difference is several orders of magnitude less than the resolution of the instrument (0.2 nm) and is about 0.4% the width of an image pixel. Considering these factors, this estimate is likely a measure of the precision to which the diffractogram spots are measured. However, a conservative estimate of the accuracy of this technique would be 1%.

Apart from the precision of determining the endpoints of a measurement line, the primary sources of error are the distortions due to the post-objective lens system. The standard distortions induced by the projection lens system are the isotropic barrel and pin-cushion distortions and the anisotropic spiral distortion, which would shift a square into what has been called a pocket-handkerchief shape. These distortions are third-order aberrations and likely have only a minor impact to the error in measuring the true distances in an image. These distortions do not cause blurring, which reduces resolution, but rather a shift or rotation of points in the image compared to the sample [10, 11].

The practicality of the technique shown here is significant. After recording a good-quality high-resolution TEM image, one simply follows the calibration procedure discussed in this article, which can be less than a minute with practice, recognizing the spots in the diffractogram.

This technique is less practical for regions of a sample that are too far from the substrate to capture both the substrate and the region of interest in the same field-of-view. Examples of this might be the following: (a) Upper metalization layers or even a gate oxide if the gate is above a "thick" strained Si lattice. (b) In FinFET devices, the top of the Fin may be too far from the substrate, and the lattice in the Fin may not be treated as having bulk lattice parameters if the surrounding layers are introducing a stress through a semi-coherent interface. (c) Silicon-On-Insulator (SOI) wafers where the SOI layer is isolated from the substrate by an amorphous insulator and not necessarily related to the substrate crystallographically.

## Summary

High-magnification, high-resolution TEM images containing regions of known crystal interplanar spacings can be used for internal calibration purposes. Through the use of a reciprocal space transformation, the non-uniform regions of the image are averaged resulting in a more accurate and reproducible calibration of the magnification of the image.

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