Gate Oxide Characterization using Annular Dark Field Imaging

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Modern integrated circuits can contain transistors smaller than 100 nm and gate oxides as thin as 2 nm. As a 0.1 nm decrease in oxide thickness can lead to an order of magnitude increase in leakage current, precise measurement of the oxide thickness is critical. We find annular dark field imaging in a scanning transmission electron microscope to be a useful method of measuring the thickness and roughness of a gate oxide. Thickness measurements are still possible in cross-sectioned samples as thick as 6000 Å. Imaging of dopant atom distributions is also possible, sometimes with single atom sensitivity.

In early 1999, a typical SiO₂ gate oxide was about 14 oxygen atoms thick. In early 2000, the oxide thickness had shrunk to 8 oxygen atoms. Between 2003 and 2008, the projected gate oxide thickness will be 4 oxygen atoms (1 nm). It is now technologically possible to produce nano-transistors with 5 oxygen-atom-thick gate oxides and 35 nm channel lengths [1]. Nevertheless, there are serious technological challenges that face the semiconductor industry if large-scale integrated circuits are to be manufactured using such devices [2]. The aggressive scaling of the gate dielectric presents challenges in controlling reliability, leakage and drive currents. The leakage due to direct tunneling through an SiO₂ gate oxide increases by roughly an order of magnitude for every Angstrom decrease in thickness [1]. The motivation for reducing the oxide thickness is to increase the electric field in the channel, thereby increasing the drive current. However as the electric field is increased above 1 MV/cm, the mobility is reduced due to surface roughness scattering. This is reflected in a decrease in drive current in transistors with gate oxides thinner than 1.3-1.5 nm for the smoothest Si/SiO₂ interfaces [1,3]. Consequently precise measurement and control of the oxide thickness and roughness is essential. Annular dark field (ADF) imaging offers quantitative measures of oxide thickness and roughness even when conventional high-resolution transmission electron microscopy (HRTEM) is beset with imaging artifacts.

A further complication is that the Si-SiO₂ interface is not atomically abrupt. For such thin devices even a 1 monolayer-thick suboxide can represent 40 % of the gate oxide thickness (since there are 2 Si/SiO₂ interfaces). Using atomic-scale electron energy loss spectroscopy (EELS) [4] and ab-initio electronic structure calculations [5, 6], it has been shown that the electrical transition region from Si to SiO₂ occurs over a region that is 0.3-0.4 nm wide, even when the structural transition is atomically abrupt. The electrical and optical properties of the interfacial region are dramatically different from the bulk properties, but can be probed using EELS [4]. Thickness measurements are still possible using annular dark field (ADF) imaging as will be discussed here.

A second set of challenges facing device designers is the positioning and activation of dopant atoms. As devices shrink, the dopant concentrations in the source and drain must increase to ensure good contact to the transistor. Such concentrations can already exceed the solid solubility limits and are rapidly approaching the 1-% level. Locating individual dopant atoms with atomic resolution is certainly within the capabilities of ADF imaging [7, 8].

In this paper we discuss the use of a JEOL 2010F, operated as a scanning transmission electron microscope (STEM), for measuring dopant distributions and gate oxide thicknesses. Electronic properties of the Si/SiO₂ interface were measured using EELS. The microscope was fitted with the analytical (C₆₀=1 mm) polypeice, JEOL ADF detector, Gatan imaging filter and BF/ADF detectors. STEM images could be recorded with both the Emispec and Gatan systems.

In a scanning-transmission electron microscope (STEM), the atomic-resolution image formed by scanning an atom-wide electron beam and collecting the signal on an annular dark field detector can also be used to align and locate the small probe needed for electron-energy-loss spectroscopy. This makes it possible to measure the chemical composition and bonding of an interface with sub-nanometer spatial resolution [4, 9-11].

Apart from the manner in which the image is formed (scanning versus parallel illumination), the main difference between ADF and HRTEM imaging is the contrast mechanism. For very thin specimens, HRTEM imaging is primarily a coherent, phase-contrast imaging technique while ADF imaging is an incoherent, amplitude-contrast technique [12-16]. Figure 1 shows the contrast transfer functions (CTFs) for ADF and HRTEM images for the JEOL-2010F-ARP. The incoherent nature of the ADF image (which measures the square of the electron wave function) largely removes the contrast reversals present in phase-contrast HRTEM. ADF imaging also trades off higher resolution for reduced contrast. Consequently, raw ADF images always look more ‘blurry’ than HRTEM images, which in turn are artificially sharpened by the microscope (The HTREM CTF removes the lower frequencies, while the ADF CTF enhances them). These models are no longer qualitatively correct for thicker specimens where multiple scattering is significant, but they still serve as a useful guide to the differences in image formation.

Figure 2 emphasizes the higher point-point resolution of incoherent imaging (roughly a factor of 40% better), showing the 1.63 Å spacing expected from figure 1. Note that antimony dopant atoms show up roughly nine times brighter than a silicon atom would - the cross section scales roughly as Z², so ADF imaging is sometimes known as "Z-Contrast" imaging [16], although not all contrast effects are related to atomic number alone. The effects of crystal orientation and strain fields can be pronounced [17], so it is always important to check the ADF result with an analytical technique such as EELS. This has been done for the case of antimony in silicon, where the ADF sensitivity is estimated to be +/- 0.5 atoms, and the EELS detection limit is +/- 2 atoms for a 100 kV VG-STEM [7]. We have succeeded in reproducing these experiments on the JEOL-
Fig.1. Contrast Transfer Functions (CTF’s) for a JEOL 2010F operated in HRTEM mode (with a 0.7 mrad beam convergence) and ADF-STEM mode (with a 1 Å diameter source, and a 10 mrad probe forming aperture). The spherical aberration coefficient of the objective lens is 1.0 mm, a typical value for an analytical polepiece. The optimal focus is chosen for each mode. Note that there are contrast reversals in HRTEM mode, but not in STEM. The dashed arrow marks the Scherzer limit of 2.3 Å beyond which information in HRTEM mode can only be recovered by image reconstruction techniques. In ADF-STEM mode, information out to 1.5 Å (the solid arrow at k=0.666 Å⁻¹) can be directly interpreted. At this point the ADF-CTF has dropped to 5% of its initial value, which is comparable to the noise levels in the image.

Fig.2. Annular dark field STEM image of an antimony delta-layer in [110] oriented Si, showing an information limit better than 0.163 nm, and minimal drift during the 40 second acquisition. The peak antimony concentration is 20 atoms or about 0.2 monolayers. (Digiscan and JEOL 2010F-ARP)

Fig.3. Bright Field STEM image of a gate oxide recorded with the Emispec system. The interface roughness was roughly 8 Å peak-peak (or 1.7 Å rms). The collection semi-angle was 1 mrad, and the probe forming aperture was 10 mrad semi-angle. (Emispec and JEOL 2010F-ARP)
2010F and find the sensitivity to be between 1 and 2 antimony atoms. Imaging As atoms has also proved practical. Light atoms, such as boron, can be imaged by reducing the ADF collector angle and imaging the strain field surrounding the dopant atoms. Single atom sensitivity for light elements in ADF is not yet possible at room temperature, but calculations suggest it should be possible with a liquid Nitrogen stage [8].

It should be noted that STEM is not exclusively restricted to ADF imaging. If a small collector (collector angle much smaller than the probe-forming aperture) is placed on axis, then the resulting bright field STEM image is formally equivalent to a conventional phase contrast HRTEM image. The collector angle in STEM plays an analogous role to the illumination angle in HRTEM. Figure 3 shows a bright-field STEM image of a gate oxide, which would be hard to distinguish from a HRTEM image.

A serious limitation to all TEM imaging methods is that the sample is viewed in projection. All information along the path of the beam is collapsed into a single intensity measurement. Roughness is imaged differently in phase and amplitude contrast images, especially in thick samples. Figure 4 shows the “black band” that is typically present in HRTEM images of Si/SiO$_2$ interfaces. (It is only in extremely thin samples that this band can be avoided). The EELS spectra on the left of the figure show that there is a substantial fraction of oxygen in the black band, as would be expected for a 6 Å peak-peak interface roughness. (This would correspond to a 1 Å rms roughness, which is typical). The “black band” is probably a more general case of dynamical diffraction artifacts seen in the roughness models of Akatsu and Odomari [18]. Their work demonstrates that HRTEM imaging can make a rough interface appear quite smooth when the film thickness exceeds the roughness correlation length (typically 10-100 Å).

In contrast, ADF images degrade quite gracefully with thickness, displaying no dynamical diffraction artifacts (provided a large detector inner angle is used). Figure 5 shows that the gate oxide thickness can be measured in specimens as thick as 6000 Å. Averaging of the roughness over the sample thickness still occurs in ADF imaging, so a rough interface will have a smooth edge, however the intensity of the bulk lattice will gradually fade away. The result is that the edge of the oxide does not appear to be well defined (since in projection it is not!). Fortunately, the incoherent nature of ADF imaging means that a line profile through the oxide will provide both the oxide thickness (from the full width at half maximum) and the roughness (convolved with the probe size). Figure 6 shows the fit of a gaussian roughness profile to each interface. In general we find a good correspondence (within 10%) between x-ray and ADF measurements of interface roughness [4].

Some caveats are in order. As a scanned probe forms the ADF images, time-varying instabilities can lead to distortions in the image (which in HRTEM would only reduce the contrast). Spatial distortions over the field of view are almost always larger than 0.2 Å. Nevertheless, the almost-incoherent nature of ADF image formation should make the method far

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**Fig. 4.** The HRTEM image recorded in the JEOL 2010F showing the 2 nm thick gate oxide and used to position the probe for spectroscopy. A Gatan imaging filter was used as the spectrometer, as the photodiode array of a conventional PEELS is not sensitive enough to detect the energy losses from a 20 pA, 0.2 nm sized probe. The interface states are visible as a pre-peak 3 eV below the O-K edge. This implies a reduced energy gap at the Si/SiO$_2$ interface [4].

**Fig. 5.** ADF images of a 2 nm gate oxide recorded at different sample thicknesses. The inner semi-angle on the ADF detector is 40 mrad and the probe forming aperture was 10 mrad semi-angle. The main effect of sample thickness is a reduction in signal/noise. The sample thickness was determined by EELS. (Digiscan and JEOL 2010F-ARP)
less sensitive to many of the problems experienced with phase-contrast HRTEM imaging. ADF images can only be formed over a much narrower range of specimen tilts than HRTEM images, reducing artifacts from misalignment of the crystal [15]. Contrast reversals with defocus are very much less likely to occur in ADF imaging, unless very large defoci or large objective apertures are used [15]. ADF images of crystals do not appear to reverse contrast with thickness as do HRTEM images [19], but they can do so at defects such as dislocations, in very thick films [17]. As in HRTEM, strain fields can complicate the image interpretation [17], but this can be minimized by using thin samples and large ADF collection angles.

In summary, ADF imaging has been demonstrated to provide quantitative information on the thickness and roughness of gate oxides. The images remain interpretable, even in thick samples. In the search for replacement gate dielectrics, the chemical sensitivity of ADF imaging is proving useful for quickly measuring reactions between silicon and the SiO₂ replacement.

References
1. G. Timp et al. (26 others), IEDM Technical Digest (1998) 615.

Fig.6. Quantitatively measuring the thickness of a 2.5 nm gate oxide from an ADF image. The gaussian interface roughness model fit is shown as a dotted line. The lower and upper interface roughnesses were 1.75 Å and 3 Å rms respectively. The inner semi-angle on the ADF detector is 40 mrad and the probe forming aperture was 10 mrad semi-angle. (Digiscan and JEOL 2010F-ARP)