

Spatially resolved vibrational electron energy-loss spectroscopy across an abrupt SiO₂/Si interface

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Recent work on the influence of an abrupt SiO₂/Si interface on the spatially resolved Si-O bond stretch vibrational signal showed a signal in SiO₂ 200 nm from the interface due to the long-range Coulomb interaction. At the interface the integrated signal intensity drops to zero. Nanometer spatial resolution is achievable while selecting the interfacial vibrational stretch signal. It is also shown that surface coupling must be considered for typical TEM sample thicknesses [1]. In this paper, we explore the experimental spatial variation of other Si-O vibrational modes as the electron probe is scanned across the interface and interpret it in terms of the relativistic dielectric theory [2].

A 3 μm layer of SiO₂ on a Si wafer was prepared for STEM EELS analysis by lifting out a focused ion beam (FIB) sample using a Nova 200 NanoLab (FEI) FIB. A NION UltraSTEM 100 aberration-corrected electron microscope equipped with a monochromator (15 meV energy-resolution) was used to perform EELS linescans across the SiO₂/Si interface. The microscope was operated at 60 kV, with probe convergence and collection semi-angles of 30 and 12 mrad respectively.

Figure 1a shows the position of the SiO₂/Si interface and the direction of the linescan in a bright-field (BF) STEM image. A typical background subtracted vibrational energy-loss spectrum observed when the probe is positioned in SiO₂ far away from the interface is shown in Figure 1b. The spectrum shows three peaks at 58, 98 and 144 meV when the probe is in SiO₂, corresponding to the thin-film Si-O bond-rocking, bond-bending and bond-stretching vibrational signals respectively [3].

Figure 2a shows the similarity in the experimental spatial variation of the 58 meV Si-O bond-rocking signal and the 144 meV signal in SiO₂, which suggests the presence of a *begrenzungs* type of effect [1]. Figure 2b shows the difference in surface contribution with change in thin-film thickness which suggests that while there is competing contribution from bulk and surface for the 144 meV signal, only surface contribution is observed for the 58 meV signal. Experimental spatial variation profiles for the bond-rocking signal as the probe moves into Si and detailed simulations to interpret the experimental energy-loss spectra in Si will be presented.

References:

1. K. Venkatraman et al., *Microscopy* (2018), p. 1-10, doi: 10.1093/jmicro/dfy003.
2. E. Kroger, *Zeitschrift für Physik* **216** (1968), 115 - 135.
3. M. Hass, *J. Phys. Chem. Solids* **31** (1970), 415 - 422.
4. The use of (S)TEM at Eyring Materials Center at Arizona State University is gratefully acknowledged.

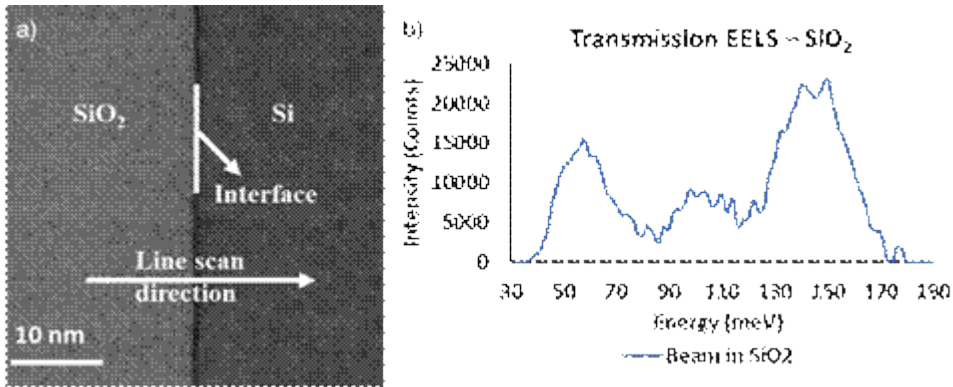


Figure 1: a) BF STEM image of the SiO₂/Si interface showing the direction of linescan. b) Energy-loss spectrum when the beam is positioned ~400 nm from the interface.

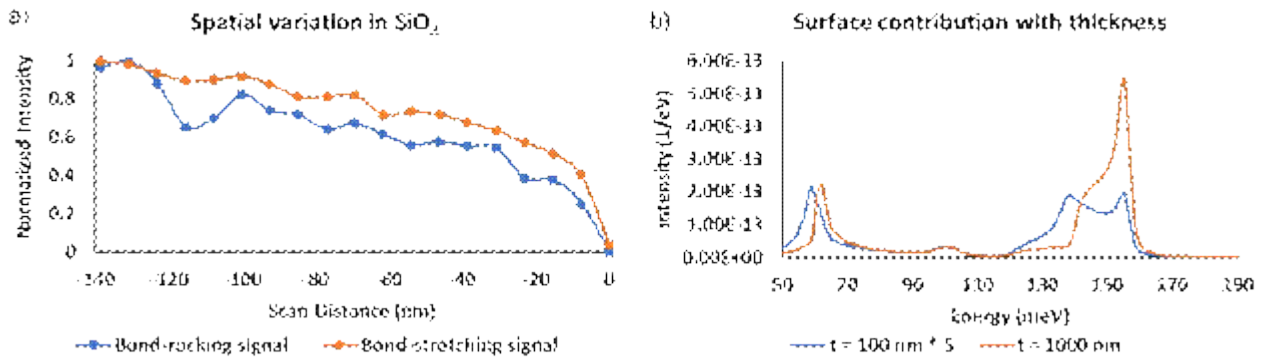


Figure 2: a) Experimental spatial variation of the 58 meV bond-rocking signal compared with that of the 144 meV signal in SiO₂. b) Variation of surface contribution with thickness calculated using Kröger's formula [2].