Atom Probe Tomography Analysis of Thick Film SiO₂ and Oxide Interfaces: Conditions Leading to Improved Analysis Yield

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Atom Probe Tomography (APT) is a time-of-flight mass spectrometry imaging technique that establishes extreme surface electric fields (~20 V/nm) on a specimen to initiate field-evaporation of surface material [1]. Consequently, it is somewhat surprising that a number of recent results have established that large band-gap dielectric materials (electrical insulators) can be analyzed by laser-pulsed APT, especially when the bulk band-gap of the material readily exceeds the photon energy of the laser-pulse used to heat the sample. These dielectric materials include but are not limited to Al₂O₃, SiO₂, ZnO, MgO, and olivine [2-6].

The analysis of native and engineered silicon oxide films of varying thickness is of interest because these structures play a critical role in device performance for current generation microelectronics [7]. Unfortunately, such structures often result in APT specimens that are prone to failure under typical analysis conditions which has undoubtedly slowed the adoption of this technique within the microelectronics industry. It is therefore essential that APT measurements are optimized, not only to ensure controlled field-evaporation resulting in data with adequate spatial and chemical resolution to impact device development, but also to maximize the potential for specimen survivability (analysis yield) to facilitate increased utility of the technique.

In this presentation, we report on our efforts to optimize APT analysis of thick film SiO₂ and Si/SiO₂ interfaces. A test structure made from a 120 nm thick SiO₂ film thermally grown on <100> silicon and subsequently capped with 300 nm of 650 °C poly-Si has been used to investigate the tradeoffs between various analysis conditions (wavelength, spot size, polarization, laser-pulse energy, evaporation rate, voltage/field control algorithm, etc.) and their effects on data quality and analysis yield. For specimens prepared in “top-down” orientation, changes in specimen temperature are monitored by variations in the Si charge-state-ratio (CSR). The most pronounced variation in CSR occurs during analysis of the poly-Si layer. Evolution of tip size and shape creates a quantum-dot-like volume of material isolated above the 120 nm thick oxide, which likely affects the photon absorption as the region evolves in size and shape. Under appropriate analysis conditions, high quality mass spectra (Si²⁺ mass resolving power FWHM of ~1200) can be achieved for the LEAP® 4000X HR (see Fig. 1). In addition, we have achieved good analysis yield (>33%) when per-pulse evaporation rates are carefully controlled through the Si/SiO₂ interface regions.

References
FIG. 1. Mass spectra and atom map (inset) for each region of the 120 nm thick oxide containing structure. Ions are color coded as shown in each mass spectrum: a) The crystal Si-substrate region exhibiting Si ions in the 1+ and 2+ charge states, b) the poly-Si region containing a small number of oxygen ions, and c) the SiO2 region exhibiting Si, O, O2, and other Si-O complex ions.