EFTEM, EELS, and Cathodoluminescence of Si Nanoclusters in Silica

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Scanning transmission electron microscopy (STEM) in combination with electron energy loss spectroscopy (EELS) and cathodoluminescence (CL) have been used to investigate Si nanocluster formation in amorphous silicon dioxide layers implanted with Si+ ions. Further on, the nanostructure of the Si doped silica films was studied by energy filtered transmission electron microscopy (EFTEM) in a 200 kV FEI Tecnai F20 TEM, which is equipped with a Gatan Tridiem 865ER imaging spectrometer. Electron transparent cross sectional samples were prepared by wedge polishing followed by low energy ion milling, see Fig.1.

Conventional bright field (BF) TEM imaging of Si nanoparticles in SiO2 can be difficult due to the very low contrast in the elastic signal. However, the contrast can be improved by imaging only with electron in a narrow energy range around the Si or the SiO2 plasmon loss peak, which are distinct in energy, see Fig.2 and 3. Using this plasmon-filtered microscopy technique in combination with tomography, Yurtsever et al. have been able to determine the three dimensional structure of Si nanoparticles in silica, [1].

The CL measurements were performed in a Zeiss DSM 960 digital scanning electron microscope with an electron beam energy Eo=10 keV and a current Io≈500 nA scanned over 100x100 µm. The CL light is collected via a parabolic mirror, a spectrograph (200-800 nm), and a charge coupled device (CCD) camera, [2].

As samples we have used amorphous, thermally grown SiO2 layers, 500 nm thick, wet oxidized at 1100 °C on Si substrate. The layers are of microelectronic quality and doped by Si+ ions with an energy of 150 keV and a dose of 5×1016 ions/cm2 leading to an atomic dopant fraction of about 4 at.% at a mean depth of about 200 nm, see Fig.1. Afterwards a post-implantation thermal annealing has been performed at temperatures Ta=700-1300°C, for 60 minutes in dry nitrogen.

Commonly, CL emission spectra of pure SiO2 are identified with particular defect centers within the atomic network of silica including the nonbridging oxygen-hole center (NBOHC) associated with the red luminescence at 650 nm (1.9 eV) and the oxygen deficient centers (ODC) with the blue (460 nm ; 2.7 eV) and ultraviolet UV band (295 nm ; 4.2 eV), [3].

In Si doped SiO2 additional emission bands are observed in the green-yellow region, see Fig.4. The CL spectra in the near IR region, Fig.5, indicate such structural changes by the appearance of an additional band at 1.15 eV associated with Si aggregates. Quantum confinement effects in these IR-CL spectra could not be observed.

References

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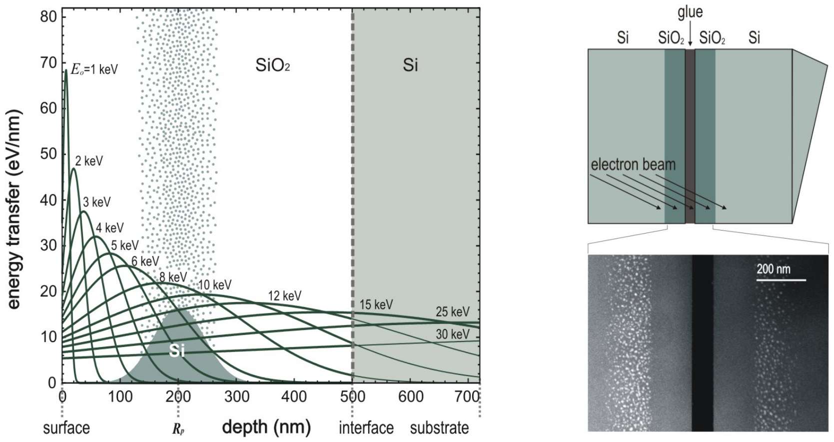

Figure 1: Si+ implantation profile in a 500 nm SiO2 layer with CL excitation densities (left) and wedge sample preparation for STEM and EELS cross section measurements (right).

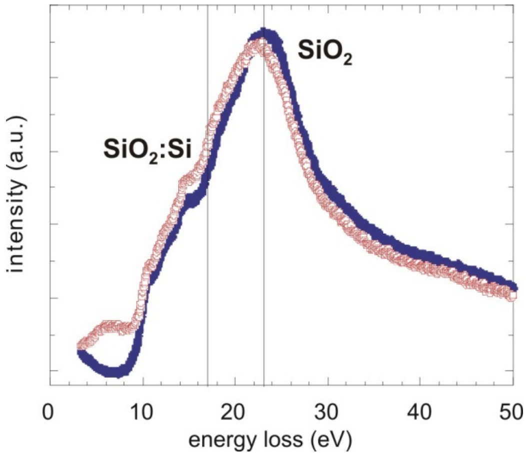

Figure 2: EELS spectra of Si+ implanted and non-implanted SiO2 regions.

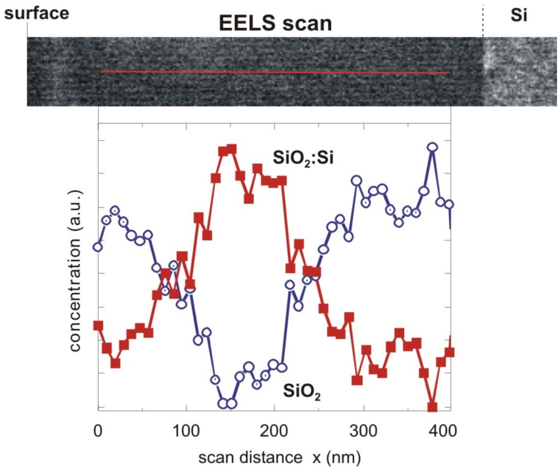

Figure 3: EELS depth profile of the Si/SiO2 plasmon losses (17/23 eV) across the SiO2:Si layer.

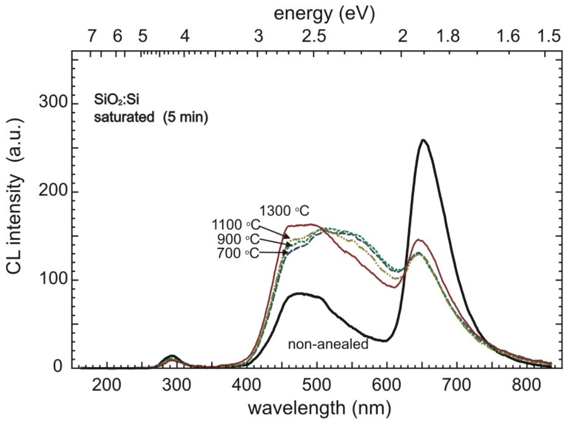

Figure 4: CL spectra of SiO2:Si layers in dependence on the annealing temperature Ta
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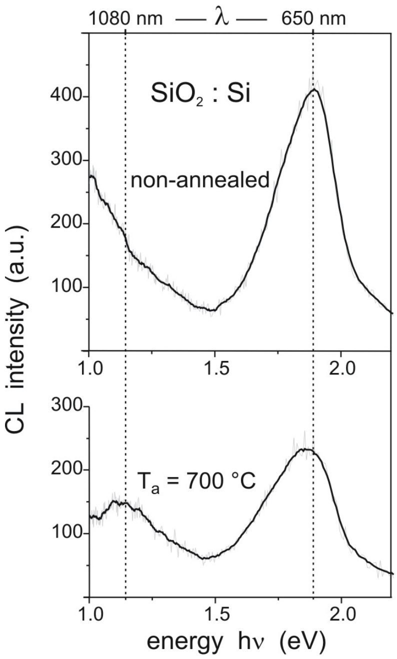

Figure 5: Near-infrared CL spectra of SiO2:Si layers without and after thermal annealing.