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EFTEM, EELS, and Cathodoluminescence in Si-implanted SiO_2 Layers

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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^+-implanted amorphous silicon dioxide layers and the formation of Si nanoclusters.
The microstructure 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 conventional wedge polishing followed by low energy ion milling.
Conventional bright field (BF) TEM imaging of Si nanoparticles in SiO_2 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 SiO_2 plasmon loss peak, which are distinct in energy, see Fig.1. 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 E_o=10 keV and a current I_o≈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 SiO_2 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 5×10^{16} ions/cm^2 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 T_a=700-1300°C, for 60 minutes in dry nitrogen. This thermal annealing leads to Si cluster formation as shown in Fig.2.
Commonly, CL emission spectra of pure SiO_2 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 SiO_2 additional emission bands are observed in the green-yellow region, see Fig.3, caused by Si atoms agglomeration up to the Si nanoclusters shown in Fig.2. The CL spectra in the near IR region, Fig.4, 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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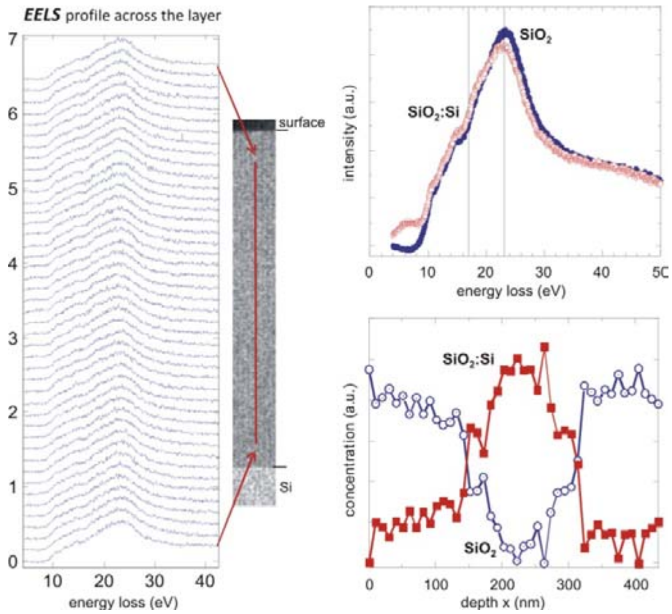

Fig.1: EELS spectra and EELS depth profile of Si/SiO_2 plasmon losses (17/23eV) across the SiO_2:Si layer.

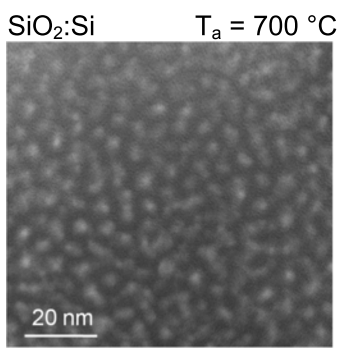

Fig.2: EFTEM micrograph of Si clusters in SiO_2 matrix using the ratio of Si/SiO_2 plasmons.

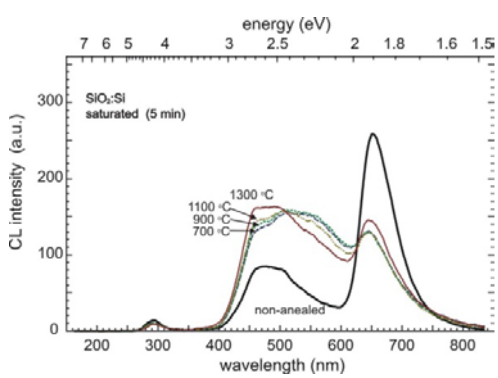

Fig.3: CL spectra of SiO_2:Si layers in dependence on the annealing temperature T_a

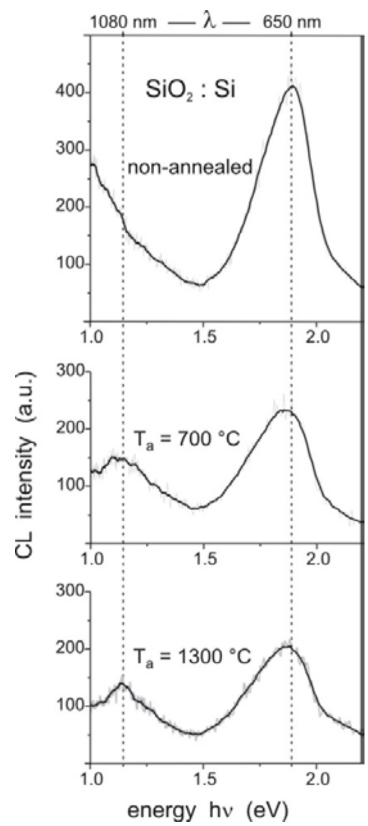

Fig.4: Near infra-red CL spectra of SiO_2:Si layers in dependence on annealing temperature T_a.

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