Cathodoluminescence of Ge- Clusters in Silica Layers

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Scanning transmission electron microscopy (STEM), energy dispersive X-ray analysis (EDX) and cathodoluminescence (CL) have been used to investigate Ge^+ -implanted amorphous silicon dioxide layers. 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), [1]. In Ge doped SiO₂ an additional emission band is identified at (410 nm ; 3.1 eV), see e.g. [2].

The CL measurements were performed in a Zeiss DSM 960 digital scanning electron microscope equipped with a liquid nitrogen cryogenic stage. The CL is excited by an electron beam energy $E_0=10$ keV and a slightly defocused current $I_0\approx600$ nA over 100x100 µm and 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 Ge⁺ ions with an energy of 350 keV and a dose of 5×10^{16} 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 $T_a = 700, 900, 1100$ °C, for 60 minutes in dry nitrogen, [2].

Fig. 2 shows the CL spectra of the Ge⁺implanted samples annealed at different temperatures T_a . The large violet emission band at 3.1 eV due to the Ge dopants is observed and the intensity of this peak increases up to a factor of 250 with increasing annealing temperature T_a up to 900 °C. Exceeding the annealing temperature to 1100°C, i.e. to the original oxidation temperature, the CL intensity is reduced again. Thus the thermal annealing process of Ge⁺implanted layers leads first to a strong increase of the violet luminescence due to formation of low-dimension Ge aggregates like dimers, trimers and higher formation; finally to destruction of the luminescence centers by further growing to Ge nanoclusters. STEM images in cross section technique [3] show the growing in Ge clusters with increasing annealing temperature, see Fig. 3. The nanocluster diameters are growing with annealing temperature from 1-6 nm at $T_a=900$ °C to 1-12 nm at $T_a=1100$ °C as shown in Fig. 4. Moreover, due to Ostwald ripening the cluster concentration drops from 4.6 to 2.6x10¹⁷ per cm³. However, their luminescent surfaces or surroundings in their sum have been reduced, i.e. the overall luminescence efficiency decreases.

References

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Fig. 1. Electron beam excitation densities in SiO₂ layers for different beam energies E_o and a Ge+ implanted profile in the mean projected range R_p =200nm.









Fig. 2. CL-spectra of thermally annealed Ge implanted SiO₂ layers measured at room temperature (RT); the respective annealing temperatures T_a are indicated.

*T*_a=1100°C



Fig. 3. STEM cross section images of Ge implanted SiO₂ layers showing clusters growing (Ostwald ripening) with increasing annealing temperatures T_a .

Fig. 4. Ge cluster diameter-distributions as a function of the annealing temperature T_a : the correlated cluster concentrations are $N_c = 4.6 \times 10^{17}$ and 2.6×10^{17} cm⁻³ for T_a =900 and 1100 °C, respectively.