The Use of the TEM as a Nanoscale Laboratory for Point Defect Creation and Agglomeration in Silicon

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Frenkel defect generation and interaction with impurities under a 200kV TEM beam have recently been observed in nitrogen doped CZ silicon [1]. The Frenkel interstitial/vacancy components remain separated due to the presence of nitrogen and/or its complexes, in lieu of recombining. The interstitial atoms, enhanced by electron collisions, diffuse away from the e-beam irradiation zone where the nitrogen stimulated complexes either capture vacancies to produce voids, or oxygen atoms to initiate SiO_2 nucleation. The benefit of this technique is that normally high temperature extended defect nucleation processes are observed at room temperature and in an area of the TEM operator's choosing.

Figure 1 provides an example of an N-CZ sample containing an ~400nm irradiated region, which has a grainy textured disturbed region, enlarged in the lower right of Fig.1, which produces the corresponding stress contours. Figure 2 shows the STEM Z-Contrast profile of the disturbed region. The foil thickness gradient is evident, as well as a reduced signal in the vacancy-rich central area. Also evident are locally higher peaks within the vacancy zone, which are attributed to nanoscale clusters. The nature of the point defect clusters has been examined more closely using HRTEM, while the oxygen profile in the irradiated area is under investigation using EELS. To further explore the role of oxygen in this process, Float Zone silicon with inherently lower oxygen concentration was irradiated and no clusters were detected.

One problem with the N-CZ samples examined is that a homogeneous distribution of the reported sample nitrogen concentration $5x10^{14}$ cannot allow more than one or two N-atoms to be in the irradiated region. A proposal was put forth that the nitrogen concentration may be locally intensified in the thin sample by the TEM preparation techniques. This possibility is being tested using SIMS measurements on N-doped wafers put through various steps of preparation. If true, the "snow plow" intensification effect could provide an easy way to measure the nitrogen concentration of lightly doped wafers, assuming that the intensification is predictable. Questions also exist about the dynamics of the interstitial migration, since the thin TEM foil sample surface can be a very strong sink for interstitials. However, using nitrogen-free CZ reference samples under identical irradiation conditions did not produce the same lateral interstitial Z-contrast segregation effects.

The effects of the TEM beam should not be limited to thin foil structural/chemical perturbations. The reaction products of this process are likely to also induce local electrical activity fluctuations. Thus, we are currently exploring e-beam irradiation to provide a signature allowing the detection of nitrogen using electronic analytical techniques, such as DLTS or EBIC. To test this, a line was irradiated on a bulk sample using the 200kV TEM beam. Schottky diodes have been deposited and irradiated/reference areas will be compared.

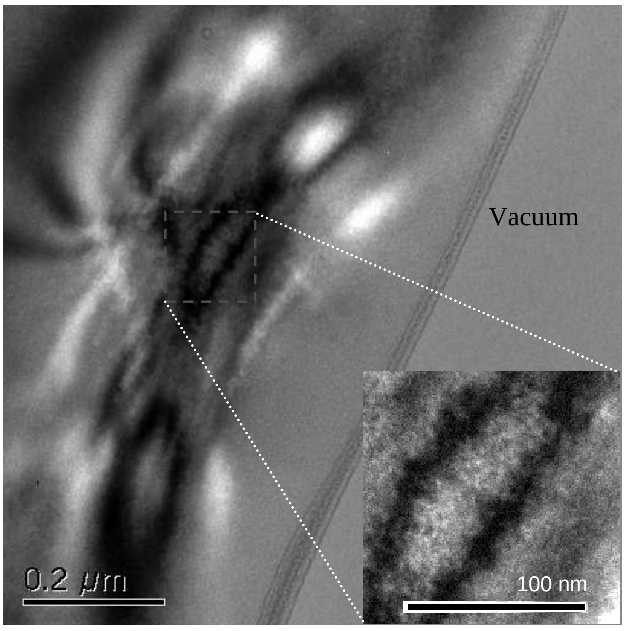

Figure 1: Conventional 200kV TEM image of e-irradiated region of N-Cz Si sample. Strong stress contrast obscures a grainy texture (inset).

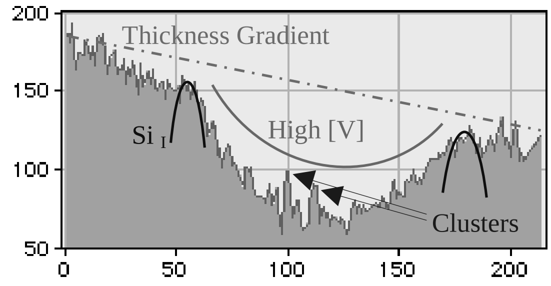

Fig. 2: Signal profile across STEM Z-contrast image of irradiated area.

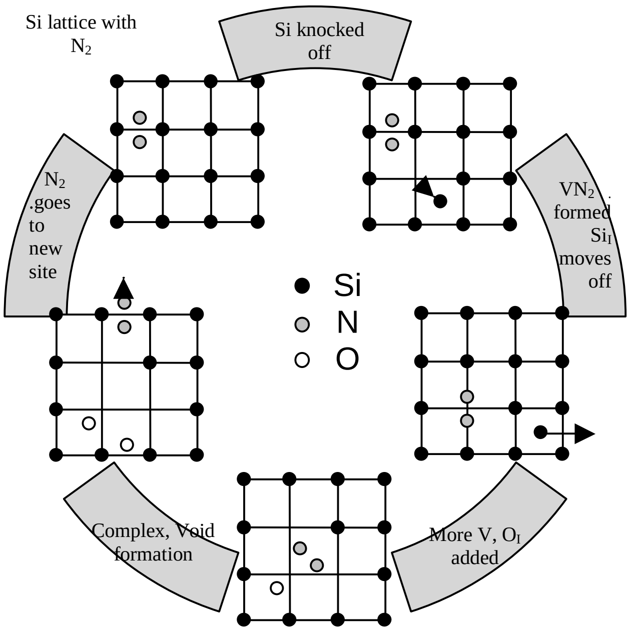

Figure 3: Cycle for SiO_2 and Void formation. In addition to N_2, V-O complexes may be active in initiating defect aggregation phenomena in N-Cz Si.

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1. N. Stoddard, A. Karoui, G. Duscher, A. Kvit and G. Rozgonyi, *ECS Proc., High Purity Silicon VII* (2002)