Photoluminescence Study on the Behavior of Self-Interstitials in Annealed Si Crystals

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1. Introduction

Oxygen precipitation takes place during annealing process of Czochralski(CZ)-grown Si crystals because of the presence of the supersaturated oxygen /1/. The oxygen precipitates produced by annealing at temperatures 600 - 1200°C consist of amorphous or crystalline SiO_2. The volume of these precipitates is roughly two times as large as that of the corresponding Si atoms in the lattice. As a result, large stresses are induced at the precipitate/matrix interface. These stresses must be relieved by the emission of Si self-interstitials or by prismatic punching of dislocation loops. This concept is well-established /1/. Actually, the prismatic punched out dislocations have been observed in CZ crystals annealed at relatively high temperatures (T > 800°C). The appearanec of extrinsic type stacking faults at annealing temperatures around 1000°C has also been explained by the same mechanism /2/. However, there have been no reports concerning the direct detection of Si self-interstitials.

The purpose of this work is to demonstrate the trace of Si self-interstitials using deep level photoluminescence (PL) spectroscopy. A strong and sharp line appears at 0.903 eV at certain annealing stages, regardless of the sources and the conductivity type of starting materials. The characteristic thermal behavior of the 0.903-eV line can be interpreted consistently assuming that the microdefects responsible for the line are correlated with Si self-interstitials.

2. Experimental

Conventional CZ-Si wafers (p-type, (100), $\rho \sim 43\ \Omega\cdot cm$, $[O] \sim 8.2 \times 10^{17}\ cm^{-3}$ (DIN), $[C] \lesssim 1 \times 10^{16}\ cm^{-3}$) are subjected to isothermal anneal at 650°C for 1 - 450 h. In order to enhance the oxygen precipitation at this temperature, the samples are preannealed at 470°C for 64 h. The PL measurement of the samples at 4.2 - 77 K is performed in the photon energy range 0.7 - 1.2 eV.

3. Results

Figure 1 shows the PL spectra of the samples at 4.2 K. We will mainly pay attention to the deep level PL ($0.7 < h\nu < 1.0$ eV). Before annealing, exciton luminescence appears in the band-edge PL but no signals in the deep level PL. An anneal at 650°C introduces a sharp and strong line at 0.903 eV as shown in Fig. 1 (b). This line consists of one main component at 0.9025 eV and four satellites with unequal spacings (Fig. 2). The intensity of the 0.903-eV line shows a distinctive annealing time dependence (Fig. 3). The intensity increases with annealing time in the initial stage, and after attaining a maximum at around 14 h it decreases gradually. If the annealing time is as long as 450 h, the 0.903-eV line becomes very weak and three broad bands appear at 0.81, 0.88, and 0.93 eV, as shown in Fig. 1 (c). The maxima of the three bands occur at the same energies as those of the so-called D1, D2, and D3 lines, respectively, which have been reported to be related to dislocations /3/.

4. Discussions

Infrared absorption studies show that the concentration of interstitial oxygen decreases with annealing time. Theoretically, one Si self-interstitial is emitted for two oxygen interstitials incorporated in the precipitate. Therefore, the concentration of Si self-interstitials increases with annealing time. However, if their concentration reaches a certain level, these Si self-interstitials begin to agglomerate, which results in the formation of interstitial-type dislocation loops. In fact, the rod-like defects, which are produced in CZ crystals by a long-time anneal at 650°C, have been identified to be interstitial-type dislocation loops /4/. Once these dislocations are formed, the concentration of Si self-interstitials decreases, since the dislocation loops grow larger by absorbing Si self-

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interstitials. Therefore, the concentration of Si self-interstitials first increases with annealing time, reaches a maximum value after a certain time, and then decreases.

The expected concentration dependence of Si self-interstitials coincides with the intensity dependence of the 0.903-eV line. This leads us to coclude that the recombination centers responsible for this line involve Si self-interstitials. The appearance of the three broad bands after a long-time anneal is also consistent with the above model, if we assume that these bands originate in the dislocations forming the interstitial-type dislocation loops. The appearance of such a fine structure as in Fig. 2 does not contradict the present model, since many sharp lines have been observed in irradiated materials where interstitials, vacancies, and their complexes are created /5/.

5. Conclusion

The oxygen precipitation in CZ-Si crystals at 650°C introduces a characteristic PL line at 0.903 eV. The recombination centers responsible for the line involve Si self-interstitials either as single interstitials or in the form of small clusters.

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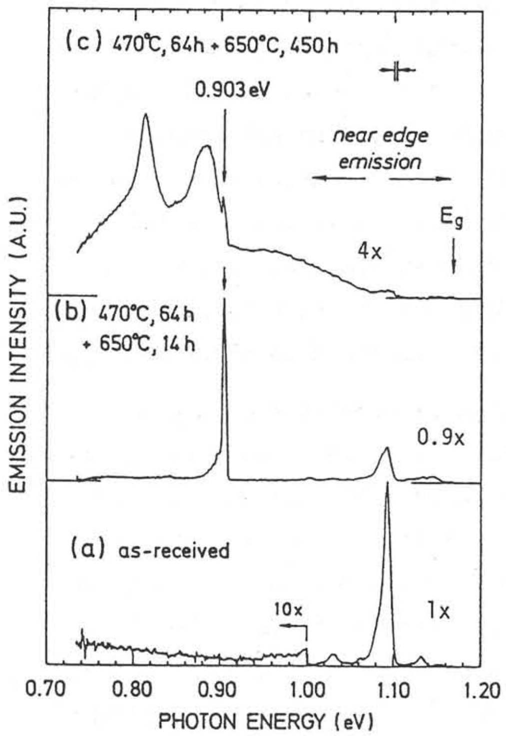

Fig. 1. PL Spectra of CZ-Si crystals at 4.2 K. (**a**) as-received, (**b**) annealed at 650°C for 14 h, (**c**) annealed at 650°C for 450 h (with preanneal at 470°C for 64 h). Spectra are corrected for the PL system response.

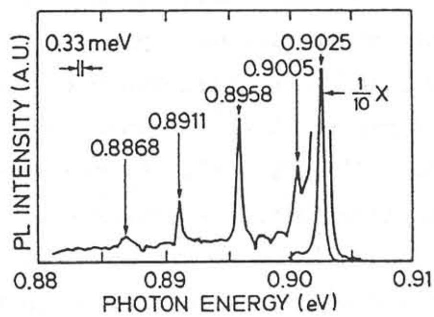

Fig. 2. Highly resolved spectrum of the 0.903-eV line. The sample is identical to that in Fig. 1(b).

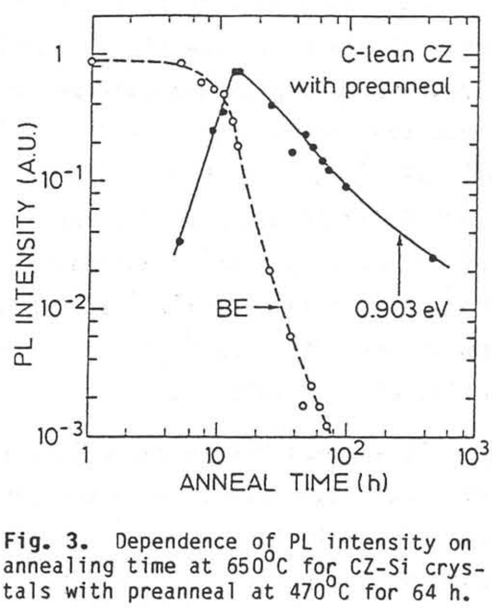

Fig. 3. Dependence of PL intensity on annealing time at 650°C for CZ-Si crystals with preanneal at 470°C for 64 h.