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Round-Robin Test of Photoluminescence Method after Electron Irradiation for Quantifying Low-Level Carbon in Silicon

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Abstract

Round-robin test of the photoluminescence method after electron irradiation was performed for the magnetic-field-applied Czochralski Si crystals with the C concentration ranging from 1.4 x 10^{14} to 3.6 x 10^{15} cm^{-3}. A universal correlation was obtained between the C concentration and the G-line intensity ratio to the band-edge emission normalized by the ratio of the reference sample. This demonstrates the effectiveness of the present method as a novel standard technique for quantifying the C impurities in the concentration range lower than 5 x 10^{15} cm^{-3}.

1. Introduction

Reliable standards for the advanced material characterization techniques are eagerly sought, since severe quality control of the starting Si wafers is required for state-of-the-art Si power devices, such as power MOS-FETs and IGBTs. The quantification of low-level C impurities is one of the most strongly desired techniques recently. Under this circumstance, Ministry of Economy, Trade and Industry (METI) is carrying out the project "Acquisition and Promotion of International Standards for Energy Saving" with one of the aims of the international standardization of the C quantification using the Fourier-transform infrared spectroscopy (FT-IR) and photoluminescence (PL) method in the concentration range lower than the current detection limit of 2-5 x 10^{15} cm^{-3} [1]. In this report we focus on the results of the preliminary round-robin test (RRT) of the PL method conducted by the National Working Group of the PL method (NWG-PL). The group consists of wafer venders, poly Si suppliers, device makers, analysis companies, equipment companies, academic experts, and administrative officers.

2. Theoretical Background

The electron irradiation in Si produces the C-related defects responsible for the G-line luminescence at 0.969 eV (1279 nm). Quantitative analysis of the C impurities has been proposed based on the positive correlations between the G-line intensity and the C concentration [2-5]. We used the intensity ratio of the G-line to the band edge emission as an index of the C concentration, following the PL standard procedure for quantifying the dopant impurities in Si [6]. Note that the G-line intensity is influenced by the O and dopant impurities.

3. Experimental Method

Samples

Forty-one samples with various concentrations of C, O and dopant impurities were offered by silicon wafer vendors and poly silicon suppliers. We selected 21 MCZ and float-zone (FZ) Si samples and irradiated with 1 MeV and 2 MeV electrons with a fluence of 1x10^{15} cm^{-2}. In this paper we report on the results of two groups of MCZ samples irradiated with 1 MeV electrons, as listed in Table I. The specifications of one group (samples E8-EB) are n-type, $\rho \approx 50$ Ω·cm, and those of another group (samples G1-G5) p-type, $\rho \geq 5000$ Ω·cm. The C concentrations of the samples were measured by secondary ion mass spectroscopy (SIMS).

PL Measurement

PL RRT was performed by 10 organizations (#1 - #10). The measurement conditions were basically in accordance with JIS and SEMI standards [6]: PL was excited by the 532-nm-laser line at 4.2 K. The measurement at 15 K and 77 K were also performed in 3 organizations.

4. Results and Discussion

PL spectroscopy

Typical PL spectra of the band-edge emission and G-line from sample G1 after 1 MeV electron irradiation are shown in Figs. 1(a) and 1(b), respectively. The G-line was clearly observed in all the samples including sample E8 with the lowest C concentration of 1.4 x 10^{14} cm^{-3} under a PL signal accumulation time of approximately 10-60 s. The band-edge emission was governed by the TO-phonon sideband of the free exciton (FE_{TO}), where the impurity bound excitons and exciton complexes were also observed (B_{TO}, P_{TO}, $b1_{TO}$). We use the intensity ratio between the G and FE_{TO} lines (G/FE) as an index of the C concentration.

C concentration dependence

The G-line intensity ratios (G/FE) reported by all the organizations were plotted against the C concentration on a log-log scale for all the samples in Fig. 2(a). We found that a positive correlation holds for all the organizations and that the correlations look very similar among the organizations. To examine the similarity of the correlations we normalized the G-line intensity ratios of the respective samples by the ratio of the reference sample (sample G1 in the present case) and plotted against the C concentration in Fig. 2(b). All the data points were on or very close to a single curve. This leads us to suggest that the correlation is universal and that the C concentration relative to the reference sample G1 can be determined using this universal curve. We believe that the current universal curve is valid for n-type MCZ samples with resistivity higher than 50 Ω·cm.

It should be noted that the PL measurement conditions were not strictly settled at the present stage. Most organizations performed the measurement at 4.2 K but some at 15 K and 77 K. In spite of the loose settlement of the conditions universal correlation was obtained. This indicates the potential superiority of the present method.

Table I Specifications of MCZ Si samples

Sample	E8	E9	EA	EB	G1	G3	G5
ρ (Ω·cm)	50-70 (*n*-type)				5000-14000 (*p*-type)		
[C] $\times 10^{14}$cm^{-3}	1.4	2.4	4.3	6.9	6.3	11	36

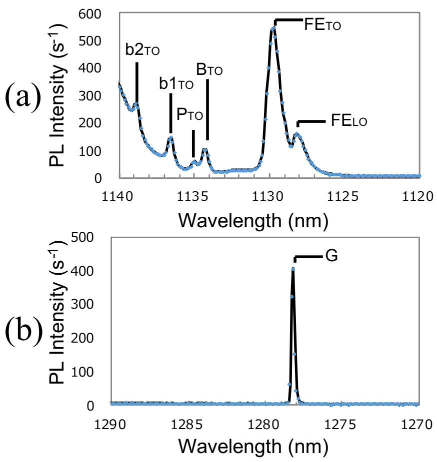

Fig. 1 PL spectra at 4.2 K of (a) band-edge emission and (b) G-line from sample G1 after 1 MeV electron irradiation.

5. Conclusions

The results of the RRT of the PL method was reported for the MCZ samples with the C concentration ranging from 1.4 – 36 x 10^{14} cm^{-3}. A universal correlation was obtained between the C concentration and the G-line intensity ratio normalized by the ratio of the reference sample. This demonstrates the effectiveness of the present PL method as a novel standard technique for quantifying the C impurities in the concentration range lower than 5 x 10^{15} cm^{-3}. The results of the FZ samples and the error analysis will be presented at the conference.

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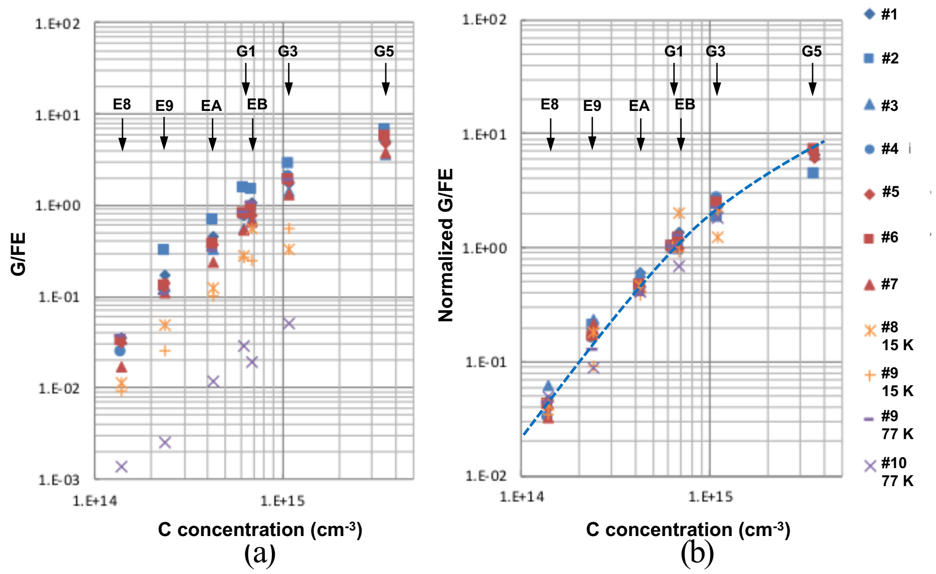

Fig. 2 Correlation between C concentration and (a) G-line intensity ratio (G/FE) for MCZ samples listed in Table I, and (b) G/FE normalized by the ratio of reference sample G1 based on PL RRT by 10 organizations (#1 - #10).