

Measurement of carbon concentration in silicon crystal

(26) Standard infrared absorption measurement procedure for industry and/or science

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シリコン結晶中の低濃度炭素の測定 (26) 赤外吸収の産業と科学のための標準測定法案

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Carbon concentration ([C]) in silicon crystal is measured by infrared absorption spectroscopy (IR) [1]. Previously, IR measurement standard has been used for quality control (QC) [2, 3]. Till 2016, new procedure for low concentration of $10^{14}/\text{cm}^3$ was developed [4] and leading companies practiced it by the round robin measurement [5]. In 2018, measurement of poly-Si and at LT was established [6]. In 2019 Si crystal replaced the prototype kilogram and C is the largest source of error of the unit [7]. Standard procedure is necessary for development (R&D) and science now. For QC, IDL (exclude type I error) is important [8]. For development and science, we introduced spectral detection limit (SDL, exclude type II error) [8]. In 2022, measurement down to $10^{13-14}/\text{cm}^3$ was demonstrated, both in single- and poly-Si and at RT and LT [9]. SIMS calibration is under examination on sensitivity and inhomogeneity in sample. The following items comprise the standard procedure for industry and/or science and single- and poly-Si.

- (1) Preparation of the reference sample with lowest and known C concentration by SIMS. Synthetic reference can be used
 - (2) Check of the instrument: Phonon band height and reproducibility
 - (3) Get single beam spectrum
 - (4) Check C absorption band width and fractional phonon bands: outer (577, 625 cm^{-1}) and inner (602, 608, 612 cm^{-1})
 - (5) Choose baseline width: Long (573-637 cm^{-1}), middle (590-618) [10], short (600-610) [11], polynomial interpolation [12]
 - (6) Create uniquely weighted differential absorption spectrum for adequate baseline (use 573-590-637 three-point baseline)
 - (7) Estimate instrumental detection limit (IDL, exclude type I error: $3 \times \sigma$ of noise)[3]
 - (8) Estimate spectral detection limit (SDL, exclude type II error, larger than the neighboring phonon bands)
 - (9) Get carbon peak absorbance larger than IDL or SDL, calculate absorption coefficient α and C concentration
 - (10) Measurement at low temperature [6]
 - (11) Measurement of poly-Si, preannealing [6]
 - (12) Report: sample: CZ/FZ/poly; C band width; baseline: L/M/S/PI; temp: RT/LT; IDL/SDL; ---
- [1] ASTM F123 (1970). [2] Inoue et al. proc. ASTM, STP960, 365 (1987). [3] ASTM F1391 (1990), SEMI MF1391-1104(2004, 2012). [4] Inoue, Phys. Stat. Sol. (c), 13 842 (2016). [5] Watanabe et al., JSAP 2016S, 20a-H113-8. [6] Inoue, Okuda, Kawamata, proc. Electrochem. Soc. 2018, 86-10, 105. [7] Ibid, JSAP 2022F, 20p-C206-8. [8] Ibid JSAP 2021F, 10p-N203-9. [9] Ibid, JSAP 2022F, 20p-C206-10. [10] JEITA EM-3503 (2002). [11] Ibid JSAP 2021S, 19ap-Z29-9. [12] JEITA EM-3512 (2009).

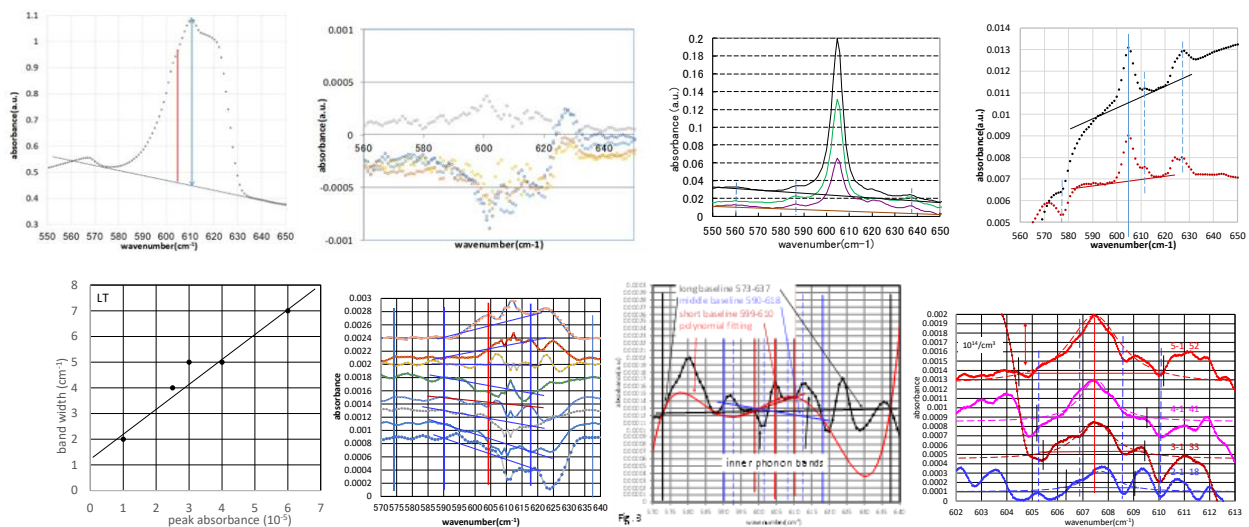


Fig. 3