

Measurement of carbon concentration in silicon crystal/ 2-nd generation

(30) Solution of inner phonon band problem in infrared absorption (2) Liquid N-T measurement

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シリコン結晶中の低濃度炭素の測定/第二世代(30)赤外吸収の inner phonon band 対策、液体窒素温度測定
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IR absorption is practical as well as scientific measurement technique of carbon concentration in Si [1]. Low temperature measurement at liquid N T was dominant especially in early days [1, 2, 3] and adopted in the ASTM Standard [4], as summarized in Tab. Four representative papers were from single and poly-Si suppliers [2, 3, 5, 6] showing the easiness of measurement at liq. N T. Moreover in 2011, measurement on the Avogadro crystal for $2 \times 10^{14}/\text{cm}^3$ was reported [7]. It established as the base of science: Carbon concentration determines the accuracy of Avogadro number and International System of Units [8]. It was said that low T measurement is advantageous for phonon overlapping problem [7].

We have established RT measurement in 1987 [9] and the results consisted ASTM Standard prepared for FTIR in 1990 [4]. In 2005 we demonstrated measurement of $10^{14}/\text{cm}^3$ at RT [10]. In 2016 we identified the middle and inner phonon band problem at RT [11]. In 2018, we reported the LT measurement of $10^{13}/\text{cm}^3$ for single crystal [12]. 5.4mm thick samples with [C] lower than $1 \times 10^{14}/\text{cm}^3$ were measured, and differential absorption spectra of all pairs are shown in Fig. Though interference fringes are observed, the inner phonon bands do not interfere for most spectra. C concentration was obtained to be 10, 9, 6, 4, $1 \times 10^{13}/\text{cm}^3$, respectively. Instrumental detection limit (IDL, 3x standard deviation of measured difference from the expected value, [4, 13]) was about $1 \times 10^{13}/\text{cm}^3$ and spectral detection limit [13] was $2 \times 10^{13}/\text{cm}^3$, for this preliminary unoptimized measurement. IR enabled $10^{14}/\text{cm}^3$ measurement of poly-Si both at RT and LT, more sensitive at LT. Ratio between the absorption at RT and LT [14] must be redetermined using FTIR. [1] Newman, J. Phys. Chem. Sol. 26, 373 (1965). [2] Baker (Dow Corning, (later Hemlock)), J. Appl. Phys. 39, 4365 (1968). [3] Endo (Komatsu), Anal. Chem. 44, 2258, (1972). [4] ASTM F-1391, SEMI MF-1391(1990). [5] Hwang (Hemlock), J. Electrochem. Soc., 138, 576, (1991) [6] Porrini (MEMC, Wacker), Solid State Phenomena, 108-109, 591 (2005). [7] Zakel, Metrologia, 48, S14 (2011). [8] International System of Units 9-th ed (2019). [9] Inoue, ASTM STP960, 365 (1987). [10] Inoue, Solid State Phenomena, 108-109, 632 (2005). [11] Inoue, Phys. Stat. Sol. C, 13, 842 (2016). [12] Inoue, ECS Trans. 86-10, 105 (2018). [13] Inoue, JSAP 2021A, 10p-N203-9. [14] Kolbesen, Solid State Electronics, 25, 759 (1982).

↔	RT↔	Low-T*↔	poly↔
1965-Newman↔	↔	8×10^{16} ↔	↔
1968 Baker#↔	↔	1.3×10^{16} ↔	↔
1972-Endo↔	1×10^{17} ↔	1×10^{17} ↔	↔
1987-Inoue↔	2.8×10^{15} ↔	↔	↔
FTIR↔	↔	↔	↔
1990-ASTM↔	5×10^{15} ↔	5×10^{14} ↔	↔
1991-Hwang#↔	↔	↔	1.5×10^{15} *↔
2005-Porrini#↔	↔	1×10^{15} ↔	1×10^{15} *↔
2005-Inoue↔	1×10^{14} ↔	↔	↔
2011-Zakel↔	↔	2×10^{14} ↔	↔
2017-Inoue↔	↔	1×10^{13} ↔	1×10^{14} ↔

