

Measurement of carbon concentration in silicon crystal

(XX) Version up of the standard Infrared absorption measurement procedure

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シリコン結晶中の低濃度炭素の測定(XX) 赤外吸収測定法規格の修正試案

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Background and purpose Carbon concentration ([C]) in silicon crystal is measured by infrared absorption spectroscopy (IR). History of representative concentration, principal paper and standard procedure is summarized in the table. Initially, the “useful range of measurement” was high. Low temperature measurement was recognized to be sensitive, and the revised standard included it and gave “the detection limit.” The first ASTM standard [1] did not define the baseline nor have the established conversion coefficient (c.c.). They were proposed from Japan (Inoue’87, [2]) and the revised standard [3] included the baseline, new c.c. and the detection limit of $5 \times 10^{14}/\text{cm}^3$. There has been no further revision since 1990, though [C] reduced much. We restarted the improvement of sensitivity meeting the demand from the power device in 2005. Mainly by the preparation of ultralow [C] reference by electron irradiation and solving machine and calibration problem by the particle based analysis, CPAA and SIMS, improved the detection limit to $1 \times 10^{13}/\text{cm}^3$. We made it possible to measure polycrystal with similar detection limit. These techniques were developed with and distributed to the Si suppliers within the global network. They were published [4, 5] and transferred to other Si suppliers who daily measure around $1 \times 10^{14}/\text{cm}^3$. Recommended procedure was established recently. Here, the SEMI standard is reexamined to include it.

	Typical[C]		Paper		Standard	Detection limit(/ cm^3)	Useful range	Ibid (LT)
1965	1×10^{17}	1965	Newman, abs	1970	F123(ver.1)		5×10^{15}	
1980	1×10^{16}	1980	Kolbesen, LT	1990	F1391(ver.2)	5×10^{14}	5×10^{15}	5×10^{14}
	5×10^{15}	1987	Inoue, conv.c.					
1991	2×10^{15}	1991	Hwang, poly	2004	MF 1391	5×10^{14}	5×10^{15}	5×10^{14}
2005	5×10^{14}	2005	Porrini, poly, LT					
2017-	$1 \times 10^{13,14}$	2005-	Inoue poly, LT	2017	(ver.3)	1×10^{13}	1×10^{14}	-

Problem, solution and reexamined parts of the standard Calculation process is illustrated in the figure.

(1) 9.1 Test sample → Both single and poly- crystals. **(2) 3.2 Carbon level of reference, 3.8 detection limit (1 Reference problem)** → Electron irradiation reduces C_s to about 1/10, supplying an ideal reference. Detection limit is improved to 1/10 of the lowest attainable $[C_s]$, $10^{13}/\text{cm}^3$ for example. **(3) 10.1 Instrumental check (2 Machine problem) (i) 10.1.3 Linearity**

→ Phonon band peak absorbance must be near 0.65, (ii) **3.8 long time and stability** → Difference between the repeated measurement of the differential absorbance spectrum must be smaller than the carbon peak absorbance. Recommended procedure: repeat 1000 scans for three times and average. **(4) 10.1.2.2 collect spectrum, (5) 10.4 obtain the carbon only spectrum (3 Phonon problem)** → Virtual phonon fractional absorption peaks appear at the inflection point of the phonon band, 600, 608 and 612 cm^{-1} (inner phonon peaks) in addition to those at 591 and 625 cm^{-1} (outer phonon peaks). **(i) 10.5 Long baseline, 10.4.2 ratio** → In case of high $[C_s]$, ratio the reference spectrum by (ref. thick./sample thick) gives the carbon-only spectrum (straight background, baseline between 573 and 637 cm^{-1} , which is the tangent to the phonon band). It is not enough for low $[C_s]$. Use the ratio to make the background at the middle point 618 cm^{-1} , where the phonon spectrum is straight, on the baseline (3 point baseline).

(ii) add JEITA Short baseline [6] → Residual background still is not straight. Short baseline (adopted by JEITA) between 592 and 618 cm^{-1} is used to determine the background absorption. **(6) 11.1 calculate the absorption coefficient, (7) 11.2, 11.3 Calculation of carbon content (4 Calibration problem)**

→ Conversion factor for the each machine is determined by measuring the block gauge calibrated by CPAA or SIMS.

[1] ASTM F123 (1970). [2] Inoue, proc. ASTM (1987). [3] ASTM F1391 (1990), SEMI MF1391-1104(2004, 2012). [4] Inoue, PSS (2016). [5] Inoue, Okuda, Kawamata, proc. ECS2018, 86-10, 105. [6] JEITA EM-3503 (2002).

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