Measurement of carbon concentration in silicon crystal (XXII) Restart of the revision of the SEMI Standard

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シリコン結晶中の低濃度炭素の測定（ＸＸＩＩ） 赤外吸収測定法規格の改訂再開

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Introduction Carbon concentration in silicon crystal started from $10^{19}/cm^3$. In the transistor and IC period, carbon affected the device performance. In the next LSI period, carbon reduced to about $10^{15}/cm^3$ and did not affect the device performance. In the 21 th Century, Si power device became popular and it uses the radiation induced CO pair for the lifetime control. Therefore, the strict control and reduction is important and the lowest level of carbon concentration reduced to $10^{13}/cm^3$ range. Carbon concentration measurement is done by the infrared absorption using the ASTM standard procedure first established in 1970. JEIDA established the advanced standard and the result was included in the ASTM revision in 1990. JEIDA was revised for the concentration reduction and the revision of ASTM started in 2014 for $10^{15}/cm^3$ regime but was interrupted. We have developed the 2nd generation measurement procedure till 2018 down to $10^{13}/cm^3$ and proposed the additional revision in 2019. The history of about 10 step activity is summarized in Tab. 1. Now the revision is restarted by the collaboration with the leading researchers and SEMI standard members over the world based on the preliminary draft of revision including 4 subjects solving the problem, **reference material, baseline, instrument test, calibration, and polysilicon measurement**. In addition, discussion on the following points started, to eliminate ambiguity and to make clear and easier procedures.

Tab. 1 History of the international collaboration and the establishment of the standard.

Year, **event**, (author), **result** (fruit), problem revealed or left

(0 1965 Newman, carbon peak identify, 1972 Endo, low temperature measurement, Nozaki CPAA calibration)
1 1970 **1st ASTM** standard (tentative), (R. Scace), baseline not established, large conversion coefficient
(1979, collaboration between NBS and NTT started (Inoue proposed to Bullis and Scace)
2 1982- 1st round robin (JEIDA) (Arai and Inoue), 1987 ASTM revision proposal (Inoue-Scace)
3 1990 **ASTM revision**, **FTIR, long baseline, small conversion coefficient**, **low T**, **detection limit**, [reference problem]
4 1994- 2nd round robin (JEIDA), **short baseline**, **small conversion coefficient**, [reference+calibration problem]
5 1997 JEIDA standard, translation of ASTM. machine and calibration problem
6 2005- International collaboration activated by the request from a car company, **synthetic reference**, (Inoue) phonon problem
7 2014- **SEMI revision** started, **short baseline**, interrupted (Watanabe) [1]
8 2014- International collaboration including **poly crystal** **cancellation of phonon bands, calibration with SIMS** (Inoue) [2, 3]
9 2015- 3rd round robin (NMS), leading companies **experienced new procedures**, (Watanabe) [4]
10 2019- **SEMI revision** restarted (Inoue) [5]

Examples of main points under discussion

1 Baseline First ASATM did not have the baseline definition. Long baseline for high concentration (560-640 cm^{-1}, over the entire phonon band) was introduced in the ASTM revision following the result of JEIDA. JEIDA introduced the short baseline (580-615 cm^{-1}, later clarified to be between the outer and inner phonon bands) for the low concentration of about $10^{15}/cm^3$ and the revision of SEMI started but is now interrupted. In the present revision, shortest baseline (600-610 cm^{-1}, on the inner phonon bands) is added for the extremely low concentration down to $10^{13}/cm^3$ range. Previous lines do not have the scientific background.
2 Wavenumber resolution It is given with ambiguity in the existing SEMI, 2cm^{-1}> for RT and 1cm^{-1}> for low T. But the measured peak absorbance may depend on it. In addition, we can only choose the nominal one, but the real one is unknown. Therefore, it must be unambiguously determined.
3 Conversion coefficient As easily understood from above, the peak absorbance may differ from machine to machine as confirmed previously. Therefore, it is necessary to calibrate individual machine by the reference material calibrated with SIMS.
4 Detection limit (DL) One important use of DL is to declare "not detected (ND)." However, there has been no serious discussion "how it is limited and how to determine the detection limit." Conventional problem of residual carbon in the reference material was solved by the synthetic reference. Noise level due to the low transmission (small signal) and the interference by the phonon

[1] K. Watanabe, draft of revision of SEMI standard (2014). [2] N. Inoue, PSSC 1-6, 201600068. [3] N. Inoue, ECS, 86-10, 105 (2018). [4] K. Watanabe, N. Inoue, JSAP 2016S 20a-H113-8. [5] Semicon Japan Srandard Meeting (Dec. 2019, Tokyo).