Measurement of carbon concentration in silicon crystal
(23) Instrumental detection limit and spectral detection limit of infrared absorption

°Osaka Prefecture University[1], °N. Inoue[1], S. Okuda[1], S. Kawamata[1]

シリコン結晶中の低濃度炭素の測定 (23) 赤外吸収法の instrumental detection limit と spectral detection limit

大阪府大研究推進 [1], ○井上直久 [1], 奥田修一 [1], 川又修一 [1]

E-mail: inouen@riast.osakafu-u.ac.jp

Introduction Second generation IR measurement technique of carbon concentration in silicon down to $1x10^{13}/cm^3$ has been established under the collaboration with leading IR researchers and Si companies in the world. We (NTT and NBS) started the collaboration with ASTM (now SEMI) in 1979 and ASTM 1990 revision was done with JEIDA [1]. To include this already and widely used technique in SEMI standard, interrupted revision was restarted in 2019. The definition, standard practice and evidence of the detection limit in the standard are not enough. In the last meeting [2], therefore, we proposed the (1) instrumental detection limit (IDL) and (2) spectral detection limit (SDL), based on the discussion with IR professionals and SEMI Standard experts. Here they are discussed again in detail and tentative examples of IDL below $1x10^{14}/cm^3$ and SDL about $3x10^{13}/cm^3$ are shown. Two other procedures were proposed then also: (3) $4cm^{-1}$ resolution, easier and more sensitive method and applicable to 1 mm thick sample [3]. (4) Background fitting function, basic procedure of spectroscopic analysis and included already in JEIDA N Standard [4], instead of baseline. They are discussed here again.

(1) Instrumental detection limit IUPAC gave a general definition and way to estimate IDL [5]: "When an instrument measures a blank sample, (99% of) the result shows empty." That is "3 times of the standard deviation of 8 measurement results." For the differential measurement, a blank sample is equivalent to 0 difference between the test and the reference. In the last meeting we showed the 8 differential spectra of the same sample for the test and the reference, with middle baseline between 590 and 618 cm^{-1} as shown in Fig.1. The standard deviation of the height of the datapoint at the carbon peak from the baseline was about $8x10^{13}/cm^3$ and the IDL was $3x10^{14}/cm^3$. Here, an example of the case for the samples of difference $3x10^{13}/cm^3$ and 0 is shown in Fig. 2. Now, the short baseline between 600 and 610 cm^{-1} is employed for [C] around $1x10^{14}/cm^3$. The standard deviation (from average difference, and 0) was $3x10^{13}/cm^3$ and the IDL was $9x10^{13}/cm^3$. Thus, the ability of IDL below $1x10^{14}/cm^3$ by the short baseline 600-610 cm^{-1} is demonstrated.

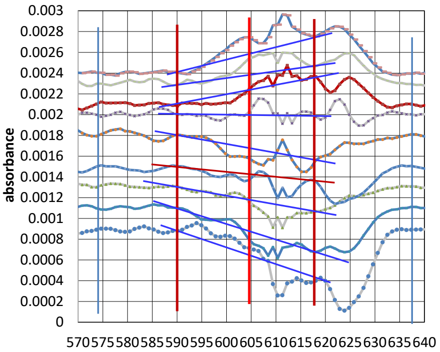

Fig. 1

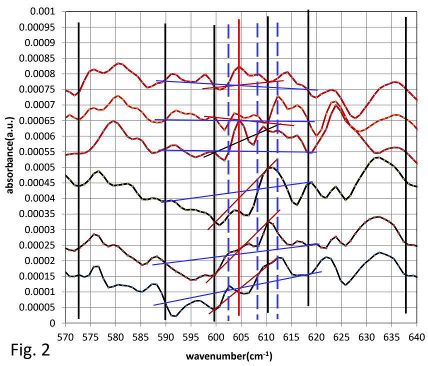

Fig. 2

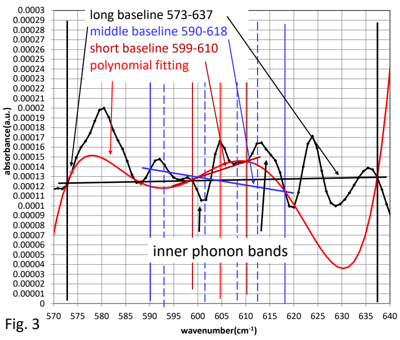

Fig. 3

(2) Spectral detection limit The instrumental detection limit is not unique but depends on the sample concentration and the baseline length. This is due to the interference by the phonon band. In real, tthe carbon absorption peak, down to $1x10^{13}/cm^3$, is distinct when the peak is separated from the inner phonon bands, as an example shown in Fig. 3. Thus, the real detection limit is defined as the height of interfering inner phonon bands. Here it is $3x10^{13}/cm^3$. It is called as the "spectral detection limit."

(3) Improvement of detection limit by $4cm^{-1}$ resolution, application to 1 mm thickness. Measurement of carbon concentration is essentially limited by the "virtual and fractional" phonon bands originating from the steep shoulders of the big trapezoid band. With a bigger resolution of 4 cm^{-1} is used, the phonon bands are reduced, resulting easy and high sensitivity measurement. Moreover, it enables the measurement of thinner samples, for example 1 mm, as already proposed [3]. The carbon peak height reduces by 10%.

(4) Fitting function instead of baseline As the carbon concentration reduces, the relative magnitude of phonon bands increases, resulting in the use of shorter baseline. Instead of the baseline, function fitting to the background, connecting the 0 datapoints (at 573, 690, 599, 610, 618 and 637 cm^{-1}), usually used in IR, is recommended as shown by the red curve in Fig. 3. It is, moreover, of course, the better estimation of the background at the peak, and given in JEITA N standard [4].

[1] Inoue dt al., ASTM STP960, 365 (1987). [2] Inoue et al., JSAP 2021 Spring, 19a-Z29-9. [3] Inoue et al., ECS Trans 2014, 64(11), 19. [4] JEITA EM-3512 (2009). [5] see website.