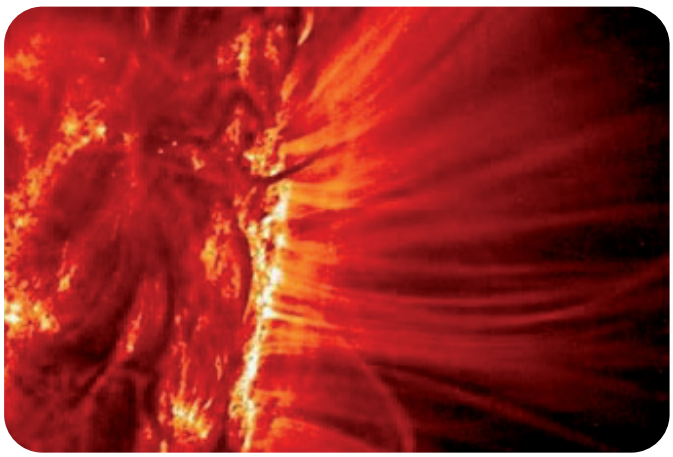

Application Note AN M54

Carbon and Oxygen Quantification in Silicon wafers

Silicon based devices, such as integrated circuits, play a key role in everyday life. Furthermore, against the background of limited fossil fuels, silicon based solar cells gain more and more in importance.

The majority of industrially produced Silicon is grown by processes (e.g. the Czochralski method), resulting in significant concentrations of interstitial Oxygen and substitutional Carbon. Depending on concentration as well as on the final application, these impurities can have both, harmful and beneficial effects. For instance the efficiency of solar cells decreases, if the Oxygen concentration is too high. On the other hand, in moderate concentrations Oxygen acts e.g. as a getter for metallic trace impurities, reducing the leakage current of the final device and makes the material less brittle. A certain amount of Carbon aids the precipitation of SiO_2 complexes which can in turn induce lattice dislocations. In general the concentration of Oxygen as well as Carbon has a fundamental influence on the electrical, thermal and mechanical properties of the final device.

Non-Destructive FTIR Analysis of Carbon and Oxygen

For the above mentioned reasons, it is essential for Silicon manufacturers to control the Carbon and Oxygen content and intense related research and development is conducted. FTIR spectroscopy is predestinated for this kind of analysis because it is sensitive, reproducible and time saving. E.g. for routine analysis the measurement duration is less than 1 minute. Moreover, FTIR based Carbon and Oxygen quantification is a non-destructive technique, giving rise to further advantages.

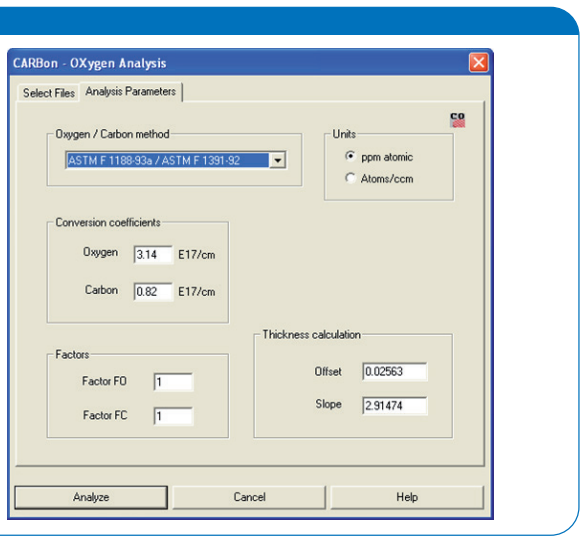

Fig. 1: Screenshot of the OPUS/SEMI software package for the comfortable determination of Carbon and Oxygen content.

The measurement is carried out in transmittance mode and the underlying principle of quantification is based on Lambert-Beer's law. This law describes the relation that for a given sample thickness the area of an absorption peak is always proportional to the concentration of the corresponding component. At room temperature substitutional Carbon gives rise to an absorption peak at ~605 cm^{-1} due to C-Si vibrations while interstitial Oxygen causes a peak at ~1107 cm^{-1} due to O-Si vibrations.

A weak Oxygen overtone at 1720 cm^{-1} can further be used for the investigation of thicker Silicon samples such as whole Silicon ingots with a diameter of up to 13''. This principle is used in Bruker SiBrickScan (SBS) analyzer system for the line mapping of Oxygen in Silicon rods. This document deals with the analysis of wafers and therefore with the classical regions at 605 cm^{-1} and 1107 cm^{-1}.

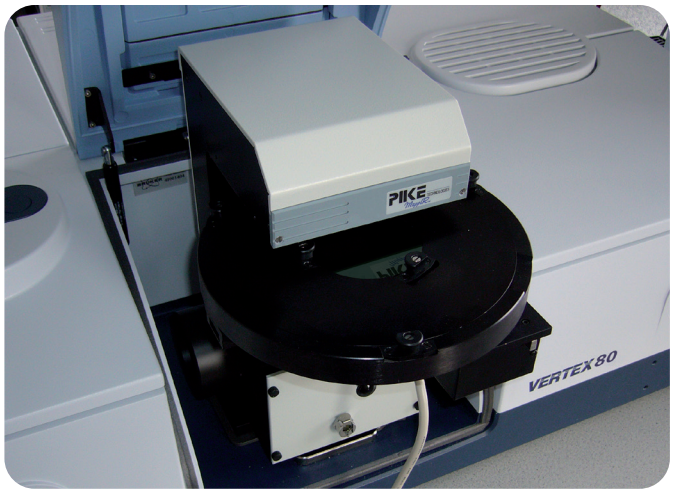

Fig. 2: Automated wafer mapping accessory in the sample compartment of a VERTEX 80 FTIR spectrometer.

In the spectral range of interest, Silicon itself shows significant absorption bands caused by lattice vibrations (so-called phonons). In order to remove these phonon contributions, the FTIR method requires a reference Si sample with negligible carbon and oxygen content, typically floatzone Silicon (FZ). For most accurate results the thickness and surface properties of the reference sample should be preferably similar to the investigated sample. Once the sample and the reference spectrum are measured, the data can be comfortably analysed using the OPUS/SEMI software package (see figure 1). All relevant evaluation standards such as ASTM F1188, ASTM F1391 and DIN 50438 are implemented in OPUS/SEMI and the user is free to choose the desired method.

In general the achievable detection limit for Carbon and Oxygen depends strongly on surface quality and thickness of the sample. Highest sensitivity is achieved with double sided polished samples. On the other hand, rough surfaces lead to scattering losses and may decrease the sensitivity and reproducibility for carbon and oxygen content. Depending on the required detection limits there are two different experimental setups which will be discussed in the following two paragraphs.

Carbon and Oxygen Routine Analysis at room temperature

Routine measurements of Carbon and Oxygen at room temperature can be carried out with any spectrometer of the VERTEX series and with INVENIO R. As long as the samples are large enough to allow for a spot size of approximately 8 mm, a room temperature DTGS detector is completely sufficient. If the lateral distribution of Carbon and Oxygen is also of interest, automated wafer mappers for the spectrometer's sample compartment are available as well (see figure 2).

In principle the sensitivity for Carbon and Oxygen increases with increasing sample thickness, but this is only valid up to a certain limit: if the sample is too thick, the silicon phonon peak will cause total absorption and at least the detection of carbon won't be possible anymore. In the case of room temperature analysis the sample thickness should therefore not exceed 2 mm. The lower limit of sample thickness is given by the onset of interference fringes which would cover the absorbance peaks of interest. Since the linewidth of the Carbon related peak necessitates a resolution of approximately 4 cm^{-1}, the sample should not be thinner than 0.4 mm. If only Oxygen is of interest, also thinner samples might be analysed. Provided that the sample properties are favourable (double sided polished, thickness of approximately 1.5mm) room temperature analysis allows for a lower detection limit in the order of $10^{16}/cm^3$ (200 ppba).

Figure 3 shows a routine measurement of a Czochralski Silicon sample (CZ) whereas floatzone silicon (FZ) of the same thickness was used as reference. Of course the OPUS/SEMI package is also able to manage the case of deviating thickness, however using a reference of the same thickness will increase accuracy. Differing surface roughness of sample and reference will result in a certain slope of the difference spectrum as it is slightly the case for the green curve in figure 3. Also this case can be handled by

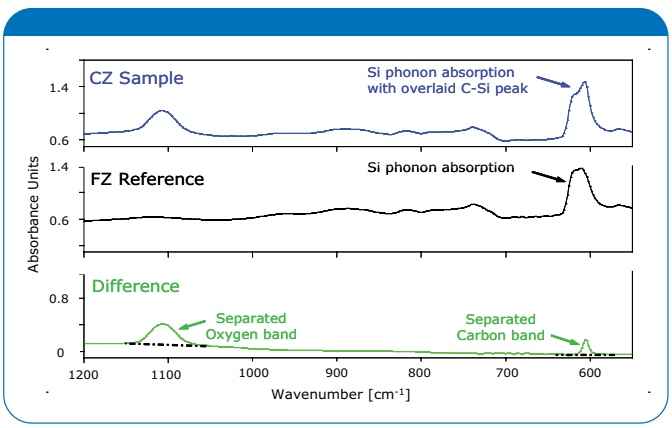

Fig. 3: Room temperature absorbance spectra of a 2 mm CZ silicon sample, a 2 mm FZ silicon sample and the resulting difference spectrum. Evaluation with OPUS/SEMI results in a Carbon concentration of $3.1*10^{17}/cm^3$ (6.2ppma) and an Oxygen concentration of $1.0*10^{18}/cm^3$ (20.1ppma). The spectra where measured using a VERTEX FTIR spectrometer with DTGS detector, KBr beamsplitter and a resolution of 4 cm^{-1}.

OPUS/SEMI applying a baseline correction as indicated by the dotdashed lines, but once again, identical surface properties are favourable. The proportionality factors between integrated band intensity and concentration (the so-called conversion coefficients) of Carbon and Oxygen are well-known from the mentioned standards and preset. However the user is free to define correction factors.

Low Temperature Analysis for improved sensitivity

Especially for Carbon the above described routine approach may not be sufficient because a lower detection limit is required. It can be significantly improved if the sample is cooled down at least to liquid nitrogen temperature (approx. 77K respectively -196°C) since low temperature analysis has two beneficial effects: the linewidth of the C-Si absorption is reduced and furthermore the intensity of the Si phonon absorption decreases, allowing for thicker samples up to approximately 4 mm (ideal thickness: 3-3.5 mm).

In case of industrial quality control for sure the CryoSAS is the ideal FTIR system for this type of low temperature analysis and fulfills all industry related requirements. In the field of research and development however, configurations based on the INVENIO R and VERTEX spectrometer series allow for more flexibility. For low temperature analysis a vacuum spectrometer such as the VERTEX 80v is highly recommended, because in a purged spectrometer residual water vapour and CO_2 absorptions tend to cover the weak signal of interest. Highest accuracies of approximately $5*10^{14}/cm^3$ (10 ppba) for Carbon can be reached with a customized liquid nitrogen cooled MCT detector in conjunction with an optical filter in order to ensure maximum sensitivity in the required spectral range. Figure 4 shows a low temperature measurement on a 3.4 mm thick Silicon sample resulting in a Carbon concentration of approximately $2.3*10^{15}/cm^3$ (45 ppba). Even in this case the measurement duration was only one minute and the sensitivity can be further improved by increasing the measurement duration respectively the number of scans.

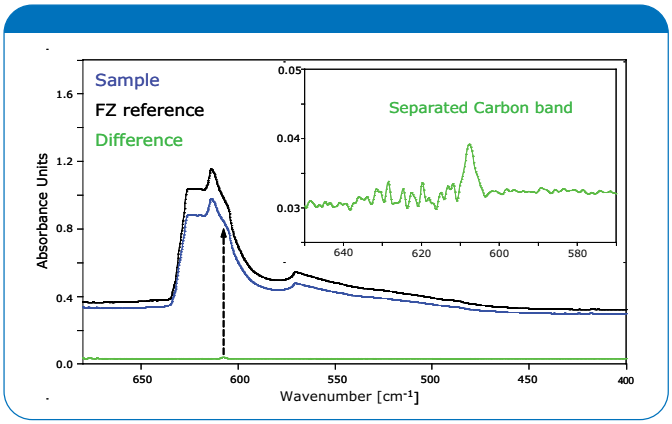

Fig. 4: Low temperature (77K) measurement of the Carbon content of a 3.4 mm silicon sample. The data were measured with a VERTEX 80v vacuum spectrometer at a resolution of 1 cm^{-1}, using a liquid nitrogen cooled MCT detector. The arrow indicates the spectral position of the weak carbon related band at approximately 608 cm^{-1}. The higher noise level on the left hand side of the separated Carbon band (see inset) is due to the intrinsic Silicon phonon absorption. Sensitivity can be further improved by increasing the measurement duration. The thickness of the FZ reference sample was slightly different which can be compensated via software.

Although a small standard cryostat with only one sample position is in principle sufficient, such a configuration is time consuming and rather uncomfortable. In order to improve the sample throughput, Bruker offers an alternative, more sophisticated cryostat with an automated sample holder for up to 6 samples, which still fits inside the sample compartment of the VERTEX 80v. All sample positions can be addressed via software control within one cooling cycle, enabling a higher sample throughput. Such a configuration is shown in figure 5: the flow through cryostat can be either operated with liquid nitrogen or liquid helium. The option of liquid helium operation has an important advantage: the same cryostat is also suitable for the far infrared quantification of shallow impurities (e.g. boron and phosphorous), which requires temperatures below 10K (see also application note 55). This means that Bruker Optics is also able to provide a high accuracy all-in-one solution for low temperature analysis of all relevant Silicon impurities.

Fig. 5: VERTEX 80v vacuum FTIR spectrometer with automated liquid N_2 or He flow through cryostat for up to 6 samples.

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