MEASUREMENT OF INTERSTITIAL OXYGEN IN SILICON INGOTS

Increased customer confidence in process control and product capability

DESCRIPTION

Interstitial oxygen content of a silicon wafer is an important material characteristic for most modern device technologies. Interstitial oxygen in silicon is typically measured by infrared absorption using either 2 mm thick slugs or thinner product wafers. The accuracy of these measurements is subject to error. These wafer measurements are time consuming and potentially introduce handling damage or contamination to the finished polished wafer. A new infrared approach allows the measurement of interstitial oxygen in single crystal silicon. Ground, large diameter, silicon crystals are profiled for interstitial oxygen using a Fourier transform infrared (FTIR) spectrometer transmitting through full diameter crystals. Measurement intervals and sample sizes may be defined prior to the wafering process, improving assurance of product quality and allowing rapid feedback to the crystal pulling floor. Whole-rod FTIR (WRFTIR) measurements can increase the producer and consumer confidence in overall process control and product capability, efficiently generating oxygen profiles along the crystal.

TECHNICAL DETAILS

Interstitial oxygen content of a silicon wafer is an important material characteristic for most modern device technologies.[1] Strengthening and contamination gettering properties of properly specified interstitial oxygen in silicon and their relationship to device performance are well understood and published.[2] Oxygen is incorporated in the silicon lattice during the growth process by dissolution of the quartz crucible.

Interstitial oxygen in silicon is typically measured by infrared absorption using either 2 mm thick slugs or thinner product wafers.[3,4] The accuracy of these slug or wafer measurements is subject to error unless both sample surfaces are polished, creating a more predictable optical transmission and internal reflection condition.[5] In addition, the crystal-pulling engineer may not fully understand the oxygen variation of the process unless most of the wafers are measured and the crystal oxygen profile is reassembled in a database. These wafer measurements are time consuming and potentially introduce handling damage or contamination to the finished polished wafer.

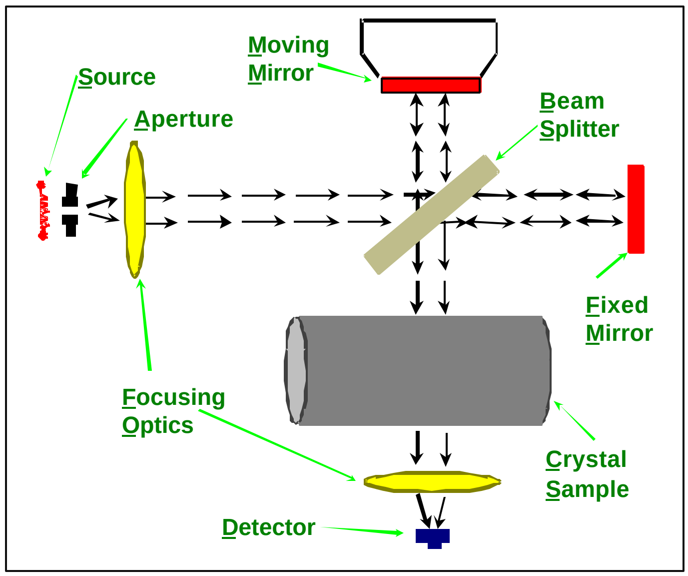

Figure 1

A new infrared approach allows the measurement of interstitial oxygen in single crystal silicon. Ground, large diameter, silicon crystals are profiled for interstitial oxygen using a Fourier transform infrared spectrometer transmitting through full diameter crystals.[6] Measurement intervals and sample sizes may be defined prior to the wafering process, improving assurance of product quality and allowing rapid feedback to the crystal pulling area. Whole-rod FTIR (WRFTIR) measurements will increase the producer and consumer confidence in overall process control and product capability, efficiently generating oxygen profiles along the crystal (Figure 1).

BENEFITS AND FEATURES

- Measurement technique increases customer confidence in overall process control and product capability
- Increases efficiency and accuracy of oxygen measurements in silicon ingots
- Allows rapid feedback to crystal engineers in the crystal pulling area
- Whole rod FTIR performance was demonstrated through direct correlation to conventional FTIR measurements

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Measurement principles

Routine measurement of interstitial oxygen in silicon wafers utilizes infrared absorption at 1107 cm^{-1} (9.03 µm). This is the absorption band associated with anti-symmetric vibration of SiO_2 in the silicon lattice.[7] The infrared beam passes through a wafer sample from the front to the back (Figure 2). An absorbance spectrum of an oxygen-free, float zone reference sample is "subtracted" from the sample spectrum to remove interference from multiple-phonon excitations of silicon near that band. Commercially available FTIR systems simulate the subtraction process in various ways for rapid measurement of the oxygen content. Quantitative evaluation of interstitial oxygen in wafers also requires accurate understanding of the measurement effects of sample thickness, surface finish, and dopant concentration. Dopant atoms like boron or phosphorus absorb infrared light and limit the usable range of infrared analysis.[8]

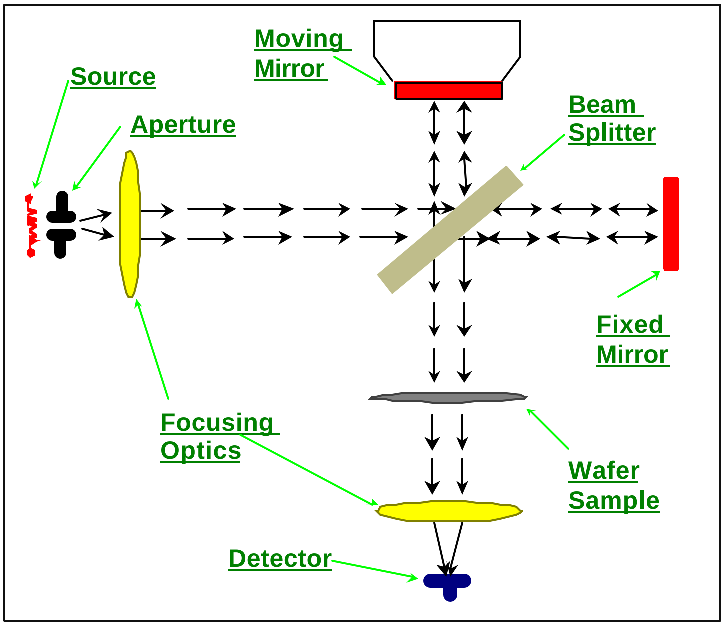

Figure 2

In the WRFTIR method, an infrared beam passes through a full crystal diameter, shown in Figure 1. The resulting absorbance spectrum represents the average interstitial oxygen content through one crystal diameter. This measurement uses a less intense, 1720 cm^{-1} (5.81 µm) absorption band that is a re-occurrence of the 1107 cm^{-1} band in silicon.[9,10] Although interference from multiple-phonon excitations of silicon is negligible near the 1720 cm^{-1} band, the band intensity is too low to be useful in wafer measurements. When measuring through a large diameter silicon crystal, however, the cumulative absorbance is enough to provide a strong measure of interstitial oxygen content with little interference.

No measurement is possible in a full diameter crystal using 1107 cm^{-1} light because almost none of it passes through the whole crystal. Likewise, absorption at 1720 cm^{-1} is too weak to provide a measurable absorption peak when the path length is a wafer thickness (Figure 3). A long path length with low absorption provides a good combination for accurate WRFTIR measurements.

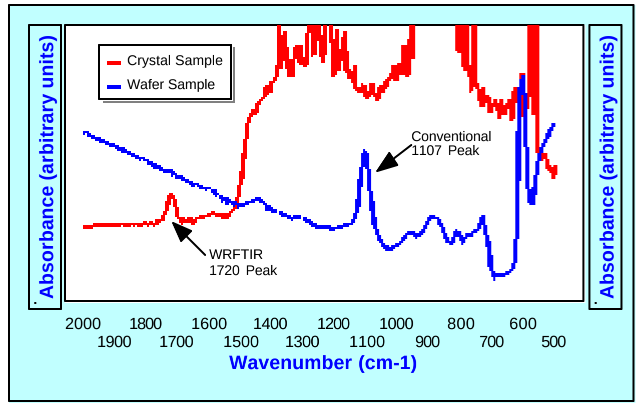

Figure 3

Instrumentation

MEMC has developed specifications for a WRFTIR system over the past few years. BioRad Laboratories further refined and fabricated the system and sells it as the QS-FRS. The instrument employs a standard 300 series optical bench mounted on a rail assembly. A mercury cadmium telluride (MCT) detector was selected for its excellent response and sensitivity characteristics.

The system is capable of measuring up to five crystal segments with a total length of 1.8 meters. These crystals remain fixed as the FTIR measures a specific point, processes data and moves to the next specified point. Collection conditions and spatial frequency of data along the crystal length are operator controlled. Instrument software allows definition of resolution, collection time, calibration, and spacing among adjacent measurement locations.

Experiment design

WRFTIR performance was demonstrated through direct correlation to conventional, wafer FTIR measurements. Sixteen p-type and four n-type, 200 mm diameter silicon crystals were selected to create a range of resistivity and oxygen values. The resistivity of the crystals ranged from 3.1 ohm-cm n-type to 56 ohm-cm p-type with the corresponding dopant density of 3.15310^{15} to 2.38310^{14} atoms per cm^3. The interstitial oxygen ranged from 11 to 17 ppma (ASTM F1188). Of these twenty crystals, growth controls for four crystals were intentionally altered to create large oxygen variations along the crystal length (Figure 4). These profile variations provided a natural dispersion in the data to be discriminated by the two methods. The full-length crystals were cut to usable lengths and ground to a nominal, 200 mm diameter.

Crystals were measured at 5 mm increments along the length and repeated twice at each defined measurement location. A single WRFTIR measurement was based on 64 scans of the FTIR mirror to calculate the absorbance spectrum. The ground crystals were subsequently processed into double side polished wafers. Wafer samples were selected at 50 mm intervals along the crystal and measured for interstitial oxygen with conventional FTIR techniques. Calibration of the WRFTIR and the conventional FTIR was performed with certified NIST

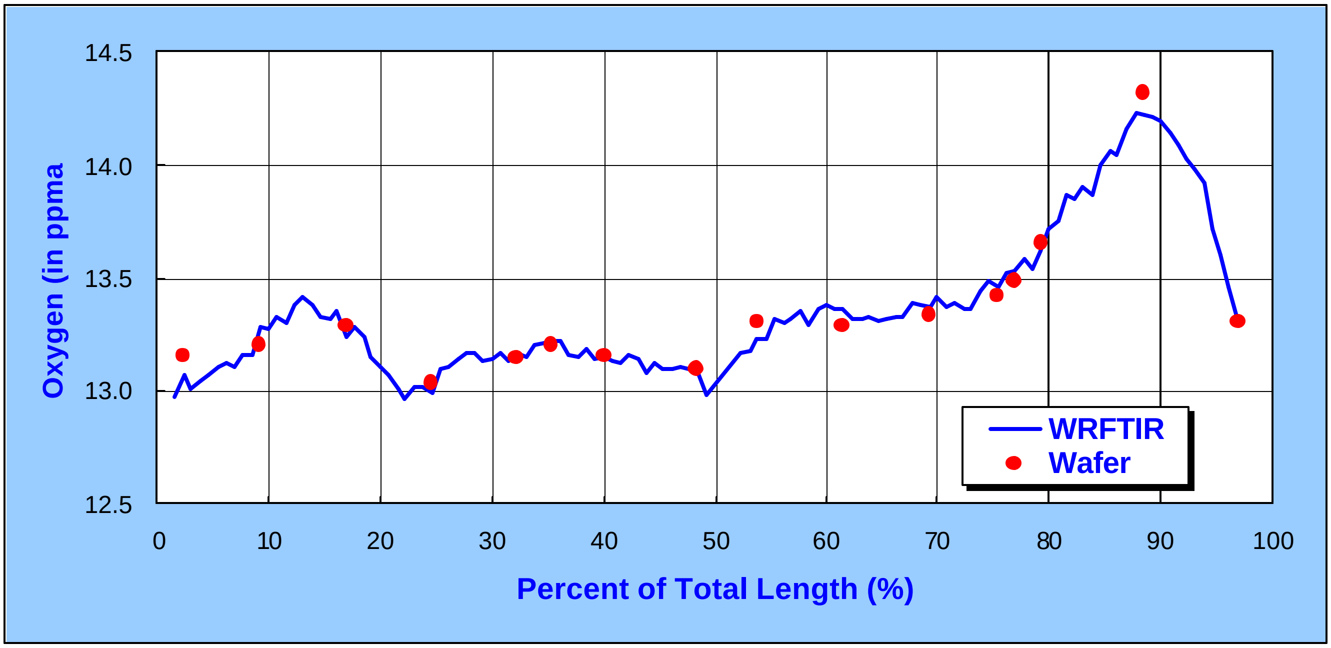

Figure 4

traceable standards.

Three different 200 mm products were selected by resistivity specifications to support a confirming production experiment. Wafer oxygen distributions, measured on random samples using conventional FTIR techniques, were compared to “simulated distributions” derived through WRFTIR analysis.

Results and discussion

Exact positions from the WRFTIR oxygen profile have been compared to corresponding double-side polished wafers selected from the crystals and measured by conventional methods. Excellent agreement was achieved between the WRFTIR and double-side polished wafer-center oxygen shown in Figure 5. The red points correspond to high resistivity (low doping) samples, and the blue points correspond to low resistivity (high doping) samples. Clearly, free carrier absorption interferes with the oxygen measurement. Wafer radial oxygen variation, considered to be a potential source of interference, is insignificant for typical oxygen gradients produced today.

Regression statistics were calculated for various subsets of the data set to demonstrate the magnitude of carrier concentration interferences. Various regression combinations of resistivity subsets (all, high or low) and wafer oxygen radial gradient subsets (all, <2% or >2%) are provided in Table 1. Each regression analysis combining high and low resistivity subsets demonstrates significantly higher standard error. Only slight degradation in correlation occurs in cases that include oxygen gradients greater than 2%.

These correlation results suggest that accurate WRFTIR calibration is possible for predicting wafer center oxygen. At

DOPANT DENSITY RANGE (xE15/cm3)	APPROXIMATE RESISTIVITY	OXYGEN RADIAL GRADIENT RANGE	QTY.	CORRELATION COEFFICIENT	STANDARD ERROR OF REGRESSION (ppma)
0.23 to 3.2	Combined	Combined	173	0.9598	0.3201
0.23 to 1.5	HIGH	Combined	148	0.9951	0.1190
1.5 to 3.2	LOW	Combined	25	0.8880	0.1720
0.23 to 3.2	Combined	<2%	88	0.9387	0.3326
0.23 to 1.5	HIGH	<2%	73	0.9940	0.1112
1.5 to 3.2	LOW	<2%	15	0.9026	0.1432
0.23 to 3.2	Combined	>2%	85	0.9709	0.3104
0.23 to 1.5	HIGH	>2%	75	0.9960	0.1213
1.5 to 3.2	LOW	>2%	10	0.7893	0.2228

Table 1

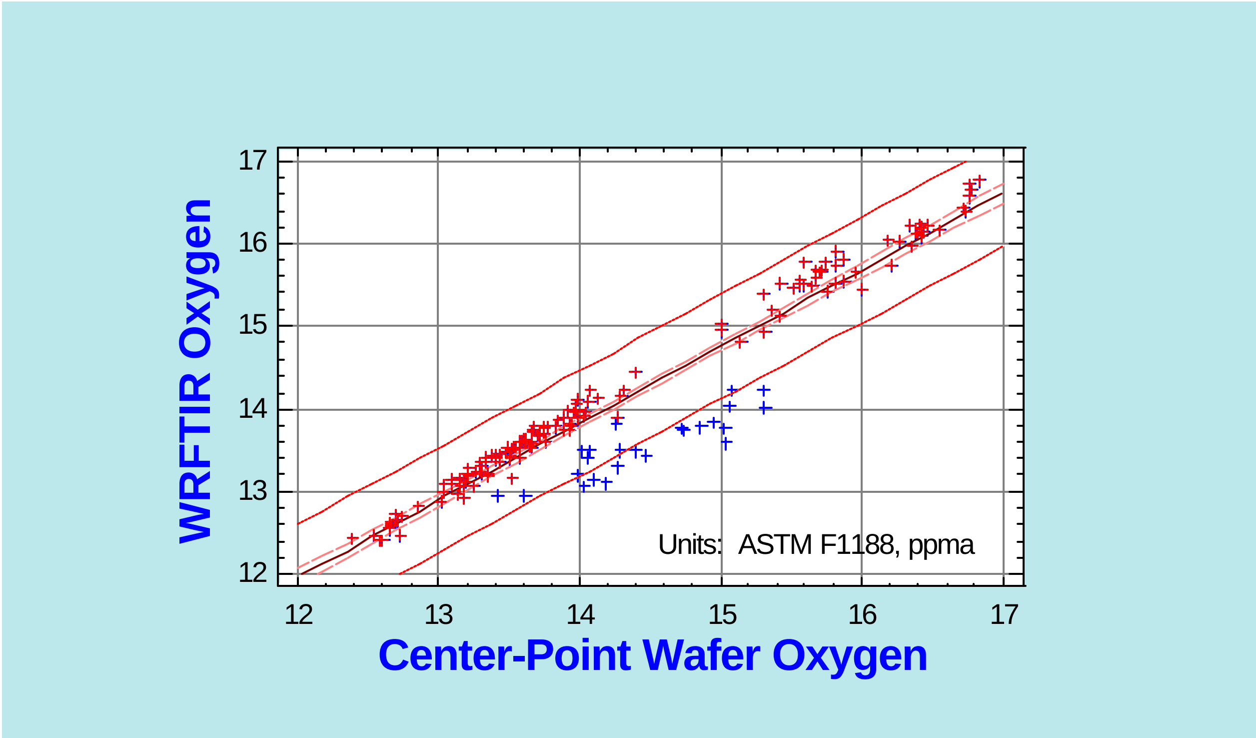

Figure 5

least two calibration options are required to assure accuracy over the normal working resistivity range. Additional WRFTIR calibration factors or algorithms for carrier concentration may be applied if subsequent testing suggests a need.

Conclusion

Average interstitial oxygen can accurately be measured in full diameter, 200 mm silicon crystals using the 1720 cm^{1} infrared absorption band. The technique provides the crystal engineer rapid feedback for continuous process improvement and control. The method gives an increased understanding of the complete oxygen distribution. Accurate calibration to NIST certified standards and routine use of the WRFTIR has been demonstrated with minimal interference from radial oxygen gradient and predictable interference from resistivity over a wide range of product specifications.

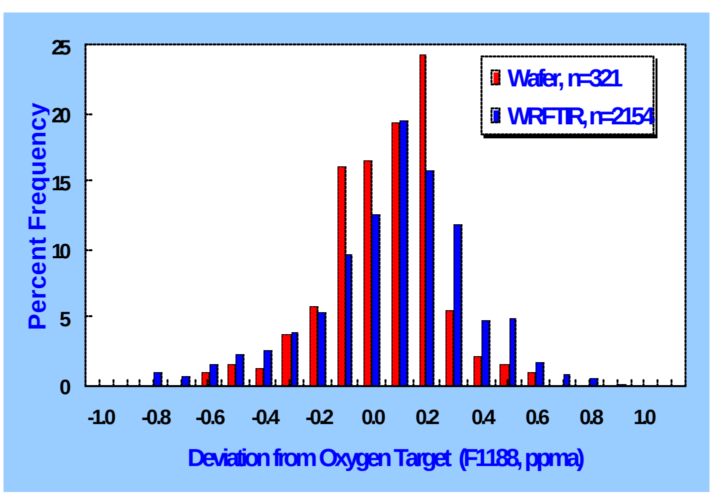

Figure 6

Acknowledgments

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