

HIGH SENSITIVITY MEASUREMENT OF NITROGEN IN CZOCHRALSKI SILICON

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A high sensitivity method for the quantitative measurement of nitrogen in Czochralski silicon by means of low temperature Fourier Transform Infrared Spectroscopy was recently presented [1], based on measurement in the far-infrared range. Its main advantage is the low detection limit for nitrogen: 5×10^{12} at/cm³. The method consists in preparing the sample by means of a thermal treatment at a suitable temperature (e.g. 650°C) for a long enough time in order to reach the saturation of the N-O concentration and therefore assure that an equilibrium situation is obtained. Under these conditions, the total nitrogen concentration can be deduced from the measured concentration of the N-O species [2]. The sample is then measured at liquid helium temperature, in the spectral range of 190 to 300 cm⁻¹, where the absorption bands related to the electronic transition of the N-O species are present. The infrared bands have been calibrated against the actual sample nitrogen concentration, which was calculated from the segregation equation, and confirmed by SIMS for those samples of concentration higher than 2×10^{14} at/cm³.

A typical infrared spectrum of a nitrogen-containing, boron-doped, CZ silicon sample showing the N-O complex-related absorption peaks at 240, 242 and 249 cm⁻¹ is in Fig. 1. The correlation between the sample nitrogen concentration and the peak area of the N-O complexes for an anneal of 30 min at 650 °C is shown in Fig. 2. The sum of the two peaks at 240 and 249 cm⁻¹ is considered here (they are the two most intense peaks, and are less interfered by the boron peak at 245 cm⁻¹). The relationship between nitrogen concentration and peak area is non-linear. This is simply due to the fact that the peak area is proportional to the concentration of the N-O complexes, which is, in turn, proportional not to the total nitrogen concentration N , but to the square root of N [2]. The calibration rule is therefore parabolic-like. The correlation is quite good: the value of the correlation coefficient R^2 is 0.98. In our experimental conditions, the minimum detectable peak area is estimated about 0.01, corresponding to a detection limit for nitrogen of about 5×10^{12} at/cm³ (0.1 ppba).

In this paper, the impact of the sample annealing temperature and of the oxygen content on the calibration equation is analyzed, and the possibility to use other N-O absorption peaks for the determination of the total nitrogen concentration is explored.

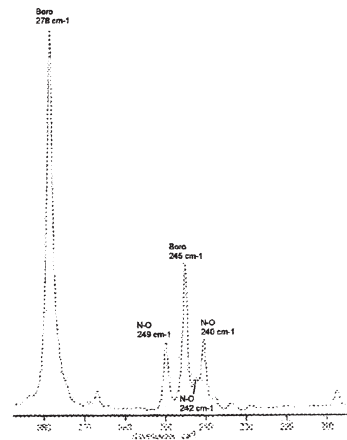


Figure 1. Typical FTIR spectrum of nitrogen containing CZ silicon sample. This sample is p-type, boron doped and contains 4×10^{14} at/cm³ nitrogen concentration. The spectrum is recorded at 10 K, under white-light illumination.

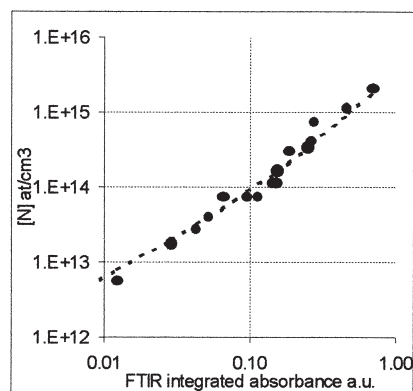


Figure 2. Correlation between the nitrogen concentration calculated by means of the segregation equation and the sum of the peak area of the 240 and 249 cm⁻¹ absorption lines. The peak area is normalized to 1mm sample thickness. The dotted line shows the 2nd order polynomial dependence of the N concentration on the FTIR absorbance

REFERENCES

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2. V.V. Voronkov, M.Porrini, P.Collareta, M.G.Pretto, R.Scala, R.Falster, G.I.Voronkova, A.V.Batunina, V.N.Golovina, L.V.Arapkina, A.S.Guliaeva and M.G.Milvidski., J. Appl. Phys 89, 4289 (2001)