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CHARACTERIZATION OF IMPURITIES IN SILICON BY IR SPECTROSCOPY

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Abstract: IR spectroscopy in various configurations (transmittance, reflectance and angular resolved) has been used to characterize, with high sensitivity and precision, different impurities which are important in the Si device technologies.

i) It is well known that the optimum oxygen concentration (from 10^{17} to 10^{18} at/cm^3), in the Czochralski grown silicon crystals improves mechanical strength, electrical properties and intrinsic gettering. Oxygen has been determined by Fourier transform infrared (FTIR) absorption in Si heavily doped with Sb (up to 5×10^{18} at/cm^3) by measuring, at room temperature, the peak height of the band centered at 1107 cm^{-1} , which arises from the antisymmetric stretch vibration of one oxygen atom bound to two neighbouring Si atoms. This has been possible, despite the high absorption background due to free carriers, by coupling the advantages typical of FTIR spectroscopy (in particular the Jacquinot and Fellgett advantages, involving an high signal-to-noise ratio) with a new procedure [1] based on thin wafers ($\approx 250\text{ }\mu\text{m}$) and short baseline ($1130 - 1080\text{ cm}^{-1}$). The results of systematic measurements on a series of 32 samples show a good linear correlation coefficient (0.97) with SIMS results and a repeatability better than 2%.

ii) In order to provide a direct correlation between grain boundaries (GB) electrical activity in polycrystalline silicon and the microscopic structure of the center(s) responsible for it, FTIR microanalysis of B-doped Si-poly, in the $4000 - 600\text{ cm}^{-1}$ wavenumber range, has been performed by scanning inter- and intra-grain regions with a $20\text{ }\mu\text{m}$ size spot. The IR spectra show a free-carrier like absorption, increasing as the spot is moved from the bulk to the boundaries of the grain, and attributed to impurity segregation [2]. This result represents the first optical evidence that electrically active impurities segregate at GB. Subsequent SIMS measurements [3], on the same samples, confirmed the segregation of boron at GB. Moreover the oxygen absorption band at 1107 cm^{-1} (the

antisymmetric stretching mode of the Si-O-Si group) results not dependent on the spot position, confirming previous SIMS results that O segregates at GB only in a very thin subsurface layer (≈ 20 nm thick) and so does not substantially influence the IR absorption.

iii) In order to study the effect of Fe impurities on B-doped polycrystalline solar grade silicon, low temperature (15 K) and high resolution (0.5 cm^{-1}) have been performed in the $230 - 360\text{ cm}^{-1}$ range. The analysis of the main boron $P_{3/2}$ series lines show a decrease of IR active boron with increasing iron (from 25 to 500 ppmw), attributed [4] to the formation of Fe-B complexes and to the accumulation of Fe at grain boundaries.

iv) Free-carrier concentration (from 5×10^{16} to $5 \times 10^{18}\text{ cm}^{-3}$) both in bulk Si and in 5–10 μm thick epitaxial layers of n-Si grown on N^+ and N^- substrates, has been determined [5] by measuring the p-polarized reflected light near the Brewster angle at $\lambda \approx 10.5\mu\text{m}$. Using a goniometer with an angular resolution of 1/400 of degree, and a CO_2 laser with a beam divergence $\leq 2.5\text{ mrad}$, we measured the refraction index variation (due to the free carriers) with a precision better than 1%, which is comparable with the best results obtained by ellipsometry. The free carrier concentration so determined agrees within 5% with results from other techniques (four-point probe, spreading resistance, C-V plots, etc.). Moreover the method allows to check the reproducibility of the substrate and of the film thickness, and the quality of the interface Si epi-substrate.

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