IMPROVEMENT IN THE SIMS MEASUREMENT OF BULK NITROGEN IN SILICON

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In 1988 the successful SIMS measurement of nitrogen in nitrogen-doped Cz-Si was first reported [1]. The typical background nitrogen levels were about $5x10^{14}/cm^3$, so the detection limit was on the order of 2 to 3 times the background level, or $1\text{-}2x10^{15}/cm^3$. In the late 1990's there was a resurgent interest in N-doping of Cz-Si at levels of $10^{13}\text{-}10^{15}/cm^3$. The ability to measure N in bulk silicon became important again but at levels often below the detection limit of SIMS. A re-evaluation of the SIMS measurement was reported in 2000 [2]. The background level was reduced to about $1x10^{14}/cm^3$, thus improving the detection limit by 5x, but still leaving an important range of nitrogen doping outside the range of detection by SIMS. Furthermore, the varying background level during a measurement posed a problem for measurement precision.

A key improvement in the measurement occurred as a result of a collaboration between a JEITA Working Group and an ASTM Task Force whereby a raster change technique, originally developed for measuring oxygen in silicon [3], was introduced to the nitrogen measurement. This technique not only resolves the varying background problem, but also greatly improves the statistics for the background subtraction [4]. A final test method was issued under ASTM F 2139-01. [5].

At Charles Evans & Associates we have achieved the following long term precisions (one relative standard deviation, RSD) using the ASTM F 2139 test method.

Doping Level	Long Term Precision (RSD)
$1.7x10^{14}/cm^3$	7%
$7.3x10^{13}/cm^3$	19%
$4.1x10^{13}/cm^3$	26%

[1] R. S. Hockett, C. A. Evans, Jr., and P. K. Chu, "The SIMS Measurement of Nitrogen in Nitrogen-Doped CZ-Silicon," in *Secondary Ion Mass Spectrometry, SIMS VI*, A. Benninghoven, A. M. Huber, and H. W. Huber, editors, p. 441, John Wiley & Sons, N.Y. (1988).

[2] R. S. Hockett and D. B. Sams, "The Measurement of Nitrogen in Silicon Substrates by SIMS," *High Purity Silicon* VI, C. L. Claeys, P. Rai-Choudhury, M. Watanabe, P. Stallhofer, and H. J. Dawson, ECS PV 2000-17, The Electrochemical Society (Pennington, NJ), pp. 584-595 (2000).

[3] A. Ishitani, K. Okuno, A. Karen, S. Karen, and F. Soeda, "Improvement of Oxygen Detection Limit in Silicon by Use of the Secondary Ion Energy Distribution and Background Subtraction," in *Proceedings of the International Conference of Materials and Process Characterization for VLSI, 1988 (ICMPC'88)*, edited by X-F Zong, Y-Y Wang, and J. Chen, World Scientific, New Jersey, 1988, pp. 124-129.

[4] N. Fujiyama, A. Karen, D. B. Sams, R. S. Hockett, K. Shingu, and N. Inoue, "SIMS quantification of low concentration of nitrogen doped in silicon crystals," Applied Surface Science **203-204**, pp. 457-460 (2003).

[5] ASTM F 2139-01, "Standard Test Method for Measuring Nitrogen Concentration in Silicon Substrates by Secondary Ion Mass Spectrometry," *Annual Book of ASTM Standards, Vol.* 10.05 (NOTE: ASTM F 2139 was transferred to the Silicon Wafer Committee of SEMI in 2004.)