Interlaboratory Calibration and Measurement Using SIMS.

Part I: Establishing a Uniform Standard Operating Procedure

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Introduction

International semiconductor companies, with offices and manufacturing facilities located around the world, require consistent analysis results regardless of which analytical laboratory acquired the data. The reliance on SIMS by the semiconductor industry to provide depth distribution information with excellent depth resolution and detection sensitivity makes interlaboratory calibration an important parameter in the specification to produce high performance devices [1, 2]. Examples of applications of SIMS in the semiconductor industry are to monitor ion implants in p- and n-channel MOS transistors [3], to qualify ion implanters [4], and to detect the presence of metal contamination in bulk silicon [5].

The goal of interlaboratory calibration is to provide reliable SIMS results that are acquired from different laboratories and instruments without compromising the technical conclusions. Measurement were made at Charles Evans and Associates (CEA) in the U.S.A. and at Evans Europa/Cascade Scientific Ltd. (CS) in the U.K. using magnetic sector mass spectrometers. In this article, the first of a series, we present results acquired without using a uniform standard operating procedure (SOP) for the two laboratories. In addition, quantification was performed using each laboratory's own set of Reference Materials acquired from different origins. This study therefore represents the most extreme case of noncompliance to accepted interlaboratory calibration practices and the minimum baseline expectation. The results obtained are compared with an internal CEA boron (bulk doped) round-robin involving four instruments using a uniform SOP.

Experimental

Both laboratories used Cameca instruments for the dynamic SIMS measurements. The samples chosen for this study were:

1. As implanted at $3x10^{15}$ at/cm^2 at 50 keV
2. P implanted at $3x10^{13}$ at/cm^2 at 10 keV
3. BF_2 implanted at $2x10^{15}$ at/cm^2 at 20 keV
4. a processed BF_2 implant.

These implant doses are expected doses and were prepared with an Applied Materials 9500xR ion implanter. The processed BF_2 sample (details not relevant to this paper) was chosen to represent a generic sample. Center portions of the wafers were diced and distributed for analysis to each laboratory. Phosphorus and arsenic high mass resolution measurements were made using a Cs^+ primary ion beam with a net impact energy of 14.5 keV. The boron measurements were made using an O_2^+ primary ion beam with a net impact energy of 8.0 keV.

Quantification was performed using relative sensitivity factors (RSF) derived from the measurements of independent (different origin) Reference Materials presently used by each laboratory for the monitored species. The depth calibration was made using a Tencor P-10 at CEA and a Tencor Alpha-Step 200 at CS. Six to eight measurements were made in one session for each sample in order to determine the precision of the analyses. All implant dose calculations were made by integrating the area underneath the implant profile starting at the

depth where a minimum is observed near the surface (or after the polysilicon layer as in the processed sample) to the background concentration of the depth profile. No background subtraction was made on any of the profiles.

Results and Discussion

Not using a uniform SOP

Table 1 summarizes the results and shows a comparison between the two laboratories for SIMS dose in atoms/cm^2, peak concentration (Cp), and projected range (Rp). The results of each laboratory show the precision of the measurements to be within ±4% RSD. This is as expected for routine measurements and may be improved to <1% RSD following a high accuracy and precision SOP -- an analytical procedure using the same sample holder window for the implant standard and unknown samples.

Table 1: Results of Interlaboratory Calibration Not Using
A Uniform Standard Operating Procedure

Laboratory	Implant Specie	Dose (at/cm^2)	C_p (at/cm^3)	R_p(Å)
CEA	As (3E15, 50keV)	2.86E15 (±0.05)	6.61E20 (±0.09)	364 (±5)
CS	As (3E15, 50keV)	3.29E15 (±0.09)	7.7E20 (±0.3)	345 (±8)
CEA	P (3E13,10keV)	2.36E13 (±0.04)	1.01E19 (±0.03)	123 (±5)
CS	P (3E13, 10keV)	2.66E13 (±0.08)	1.03E19 (±0.04)	121 (±4)
CEA	BF_2 (2E15, 20keV)	1.85E15 (±0.02)	7.7E20 (±0.1)	183 (±5)
CS	BF_2 (2E15, 20keV)	1.65E15 (±0.02)	6.4E20 (±0.1)	169 (±4)
CEA	Processed Boron	6.0E12 (±0.2)	4.02E17 (±0.04)	2590 (±20)
CS	Processed Boron	6.76E12 (±0.09)	4.2E17 (±0.1)	2670 (±20)

A comparison of the accuracies between the two laboratories for the dose, Cp and Rp are presented as values (% RSD) in Table 2. The average dose value of 6.2% RSD is within the accuracy variation of 10% RSD expected for *independent measurements.* Our observed value of 6.2% RSD is much less than the 15.8% RSD variation reported for a concensus phosphorus round-robin exercise involving nine independent laboratories. Quantification in that phosphorus round-robin was made using implant standards specific to each laboratory [6].

Table 2: Interlaboratory Accuracy Not Using
A Uniform Standard Operating Procedure

Implant Specie	Dose (%RSD)	C_p (%RSD)	R_p (%RSD)
As (3e15, 50keV)	6.9	7.1	2.6

P (3e13, 10keV)	5.9	1.2	0.6
BF_2 (2e15, 20keV)	5.7	9.2	4.1
Processed Boron	6.2	1.8	1.6

The fact that a dose value of 6.2% RSD is observed in this study indicates that the independent protocols are similar and that the implants used for calibration at CEA and CS have similar nominal dose accuracies.

Using a uniform SOP

Accuracy variations may be improved to <5% RSD by using a uniform SOP, to normalize instrument conditions such as sputter rate and crater depth measurement, and using identical Reference Materials for calibration. For example, following an SOP for *routine, rapid throughput analysis*, and using certified multi-window sample holders (maximum of 12 windows), an accuracy variation of <3% RSD can be expected. This variation may be attributed to the non-uniformity of the sample holder face-plate [7].

Table 3 shows the result of an intralaboratory CEA boron (bulk doped) round-robin study involving four instruments and indicates that an accuracy variation of ±0.90 % RSD is possible using an SOP for *high precision, rapid throughput* analysis. It has also been demonstrated at CEA that using a high accuracy and precision SOP (*high precision, low throughput*) analysis, a reproducibility of <0.5% RSD can be achieved.

It should be noted at this point that the analysis of these samples using a magnetic sector mass spectrometer does not necessarily represent the best analytical technique for measuring ultra-shallow implants. Depending on the information required, TOF-SIMS and Quadrupole SIMS may be preferred techniques for acquiring accurate dose and distribution information of ultra-shallow implants [8].

TABLE 3: Results of intralaboratory calibration using a high precision, rapid throughput uniform standard operating procedure.

Instruments	Results (measurements acquired on same day)	**Average**	**%RSD**
A	1.298, 1.309, 1.261, 1.198, 1.171, 1.178E19	1.239E19	5.0
B	1.205, 1.209, 1.204, 1.207, 1.216, 1.210	1.209E19	0.3
C	1.217, 1.225, 1.232, 1.223, 1.242E19	1.228E19	0.7
D	1.237, 1.238, 1.250, 1.244, 1.224, 1.231E19	1.237E19	0.7
Intralaboratory Result: Average = 1.228E19; 0.90 % RSD			

Conclusion

This study shows that the selection of rapid throughput analytical conditions does not compromise accuracy of results when an SOP is followed and identical Reference Materials and certified sample holders are used. With several standard operating procedures available at CEA, the appropriate cost effective analysis can be performed to comply with the client's requirements. We anticipate that a *routine* high precision, high throughput interlaboratory accuracy of less than or equal to 3% RSD between CEA and CS can be achieved *using uniform SOPs, identical Reference Materials, and certified sample holders.* Continued efforts to improve our interlaboratory accuracy are motivated by the fact that each laboratory independently services a major portion of the semiconductor industry, in different geographic regions.

References

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