

Quantitative Measurement of Arsenic Implant Dose by SIMS

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Issues associated with making quantitative measurements of the arsenic implant dose in silicon by SIMS are described. These include the use of a certified reference material for calibration, the choice of silicon matrix reference species, the matrix normalization method, and minimization of detector count losses. A round-robin study is being conducted by ISO TC201/SC6 to determine the best analytical procedures and the level of interlaboratory agreement for this type of measurement.

INTRODUCTION

The International Organization for Standardization Technical Committee 201, Subcommittee 6 is responsible for creating documentary standards for secondary ion mass spectrometry. One current activity of the subcommittee is the development of a method for depth-profiling of arsenic in silicon. This is a logical step that follows the issuance of ISO 17560 – Method of depth-profiling of boron in silicon.

Some of the subjects that must be addressed in a documentary standard are the method of calibration and the specific technical issues for each measurement system. For the case of depth-profiling of arsenic in silicon by SIMS, the issues related to quantitative implant dose and concentration measurements that have been identified include the choice of silicon matrix reference species, the matrix normalization method, and minimization of detector count losses. These topics will be considered in turn in the following sections.

CALIBRATION METHOD

Access to a certified reference material (CRM) is important both for obtaining traceability to the International System of Units (SI) and for helping to achieve interlaboratory comparability. A 1997 round-robin study of arsenic implant dose measurements that was conducted in the United States before a CRM became available showed poor

interlaboratory agreement, with results varying by 30% even after a large outlier was excluded. These results were attributable to the lack of a common reference material. Since that time, SRM 2134 has become available from the U.S. National Institute of Standards and Technology [1]. It is a 100 keV ion implant of arsenic in silicon with a dose uncertainty of only 0.38% at the 95% confidence level. A SIMS depth profile of this material is shown in Fig. 1.

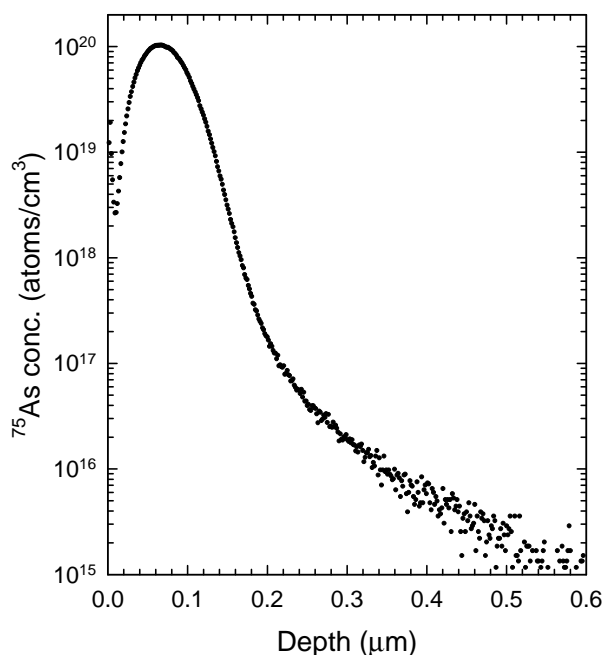


Fig. 1 SIMS depth profile of As in SRM 2134 [2].

The SIMS profile of a reference material is used in the calibration process to calculate a relative sensitivity factor (RSF) as described in ISO 18114 – Determination of relative sensitivity factors from ion-implanted reference materials.

CHOICE OF MATRIX SPECIES

According to ISO 18114 the RSF for isotope j of species A with respect to isotope k of matrix M is calculated from the depth profile of an implanted reference sample using the following equation:

$$RSF^{A_j, M_k} = \frac{\Phi \times n}{\sum_{i=1}^n \left[\frac{(I_i^{A_j} - I_{BG})}{I_i^{M_k}} \right]} \times d, \quad (1)$$

where Φ is the implanted fluence (dose), n is the number of profile cycles, d is the profile depth, $I_i^{A_j}$ is the count rate of isotope A_j at cycle i , $I_i^{M_k}$ is the count rate of reference isotope M_k at cycle i , and I_{BG} is the mean background count rate of species A_j . The concentration of the same analyte in an unknown sample of the same matrix is determined from the RSF according to Equation 2:

$$C_i^{A, M} = \frac{RSF^{A_j, M_k} \times I_i^{A_j}}{I_i^{M_k} \times N^{A_j}}, \quad (2)$$

where N^{A_j} is the fractional isotopic abundance of isotope A_j . Thus, the quantification procedure depends on specific choices for both analyte species and matrix species. The matrix species is normally selected to minimize variability in the RSF due to analysis conditions that are difficult to control such as tilt or height variations of sample holders and differences in analysis locations with respect to mask edges. In practice these variations can be minimized by selecting a matrix species with energy and angular distributions that closely match those of the analyte. An accepted rule for SIMS has been that this condition is achieved by matching an atomic analyte species, e.g., As^- , with an atomic matrix species, e.g., Si^- , and a dimer analyte species, e.g., $AsSi^-$, with a dimer matrix species, e.g., Si_2^- . The most sensitive detection of arsenic in silicon by SIMS is achieved by using Cs^+ bombardment and $AsSi^-$ detection, so Si_2^- would normally be selected as the reference species

according to this rule. However, a recent study suggested that an Si_3^- reference produced lower variability in the arsenic RSF determination [3]. A round-robin study is underway to test the generality of this observation.

Because silicon has 3 stable isotopes, its dimer and trimer clusters contain 5 and 7 isotopic species, respectively. Any of the species within a cluster should have the same effect on RSF variations when used as a matrix reference. However, other considerations may dictate the choice of one species over another within a cluster. Figure 2 shows a plot of $AsSi^-$ and 4 negative silicon dimer species with m/z from 57 to 60 from an arsenic ion implant with a dose exceeding $1 \times 10^{16}/cm^2$. The Si_2^- profiles with m/z 59 and 60 exhibit surface peaks that are not seen with the other two dimer species. These peaks must be due to spectral interferences from undetermined sources and argue against the use of either of these isotopic species as a matrix reference. However, the count rates at m/z 57 and 58 are considerably higher and their use may lead to undesirable detector saturation effects.

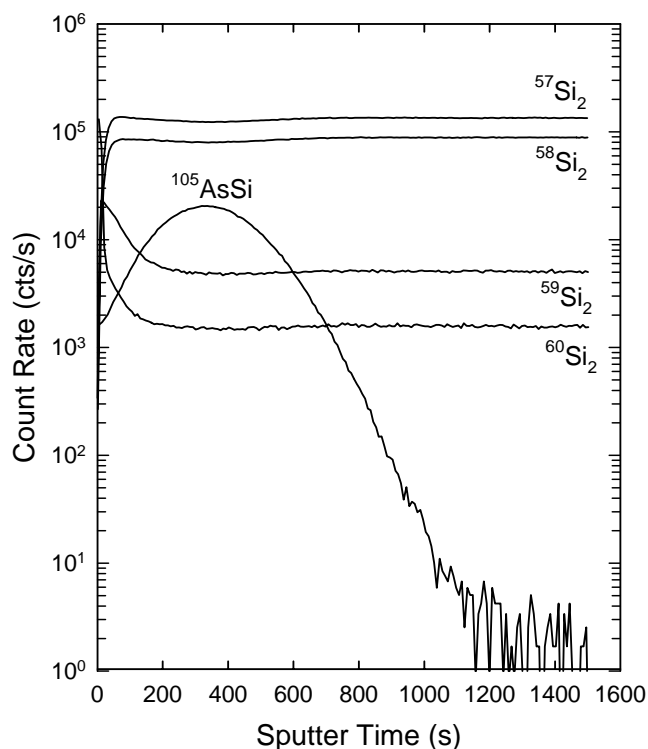


Fig. 2 SIMS depth profiles of high-dose arsenic implant and four possible choices of Si_2 matrix species.

MATRIX NORMALIZATION METHOD

ISO 18114 allows a choice between two matrix normalization methods. Either a single average value can be used for the matrix species count rate or the analyte count rate can be divided by the matrix count rate on a cycle-by-cycle basis as indicated in Eqs. 1 and 2. When the matrix count rate is constant, the choice of normalization method will have little influence on the result. However, for high-dose arsenic implants such as the one shown in Fig. 2, the matrix signals are reduced in the depth region of highest arsenic concentration. In this case the choice of matrix normalization method makes a significant difference on the calculated arsenic dose and concentration profile using the RSF method. A recent round-robin study conducted by Japanese SIMS users in the JSPS-141 SIMS Depth Profiling Working Group suggested that cycle-by-cycle normalization can extend the linearity of the RSF approach to arsenic implant doses as high as $3 \times 10^{16}/\text{cm}^2$ [4]. Several issues still remain to be tested. One is whether the same conclusion will hold if Si_3^- is used for matrix normalization. Another is to confirm the doses of the high-dose implants by independent measurements. These points will be covered within the scope of a new round-robin study.

DETECTOR COUNT LOSSES

In pulse-counting ion detection systems, count losses occur when two ions arrive within the dead time of the counting system because only the first one will be counted. When performing depth-profile analyses, SIMS systems are gated to reject secondary ions that originate near the edge of the sputtered crater. Gating for crater edge rejection can be accomplished through the ion optics by the placement of a physical aperture in an intermediate focal plane, or through the use of gating electronics synchronized with the primary beam raster scanning system. In either case, the effect of the gating on a raster-scanned system is that the count rate averaged over the scanned area will be less than the count rate when the gate is open by a factor that is roughly equal to the gated area divided by the total scanned area. This effect can lead to significant count losses even though the average count rate may not seem to be

excessive. The fraction of counts lost is to first order equal to $n \cdot \tau$ where n is the *instantaneous* count rate and τ is the system dead time. For example, consider a system with a 20 ns dead time and a 5% gated area. If the raster-averaged count rate at the peak of an implant is 5×10^5 cts/s, the instantaneous count rate when the gate is open will be 1×10^7 cts/s resulting in about a 20% loss of counts. One must always be aware of this effect when attempting to make high accuracy implant dose measurements because most SIMS data systems do not take counting losses of this type into account.

An option that is available when depth-profiling arsenic implants using AsSi^- as the analyte species is to monitor the $^{75}\text{As}^{30}\text{Si}^-$ signal for higher dose samples because it will have about 30 times lower count rate than the $^{75}\text{As}^{28}\text{Si}^-$ signal, as illustrated graphically in Fig. 3 [5]. This technique can avoid significant count losses at the peak of an arsenic implant and extend the range of linear coverage of the SIMS measurement. If the arsenic profile in the reference sample is monitored as $^{75}\text{As}^{28}\text{Si}^-$ for the RSF determination, the RSF correction factor should be determined empirically

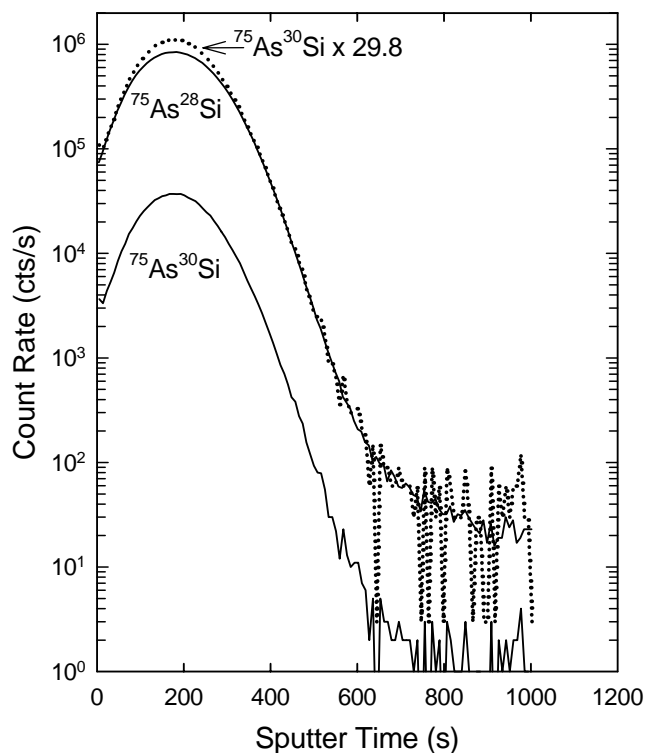


Fig. 3 Count losses at the peak of a high-dose As implant monitored with $^{75}\text{As}^{28}\text{Si}^-$ can be avoided by monitoring $^{75}\text{As}^{30}\text{Si}^-$.

from the ratio of integrated counts in the m/z 103 and m/z 105 profiles under conditions where the m/z 103 profile loses negligible counts due to dead-time effects at the peak. This ratio should be close to the expected ratio of 29.8 for the natural isotopic abundances of ^{28}Si and ^{30}Si , but it may be different due to isotopic discrimination effects in the SIMS system.

Similar count loss effects apply to matrix species as well as implant species, and should also be taken into account as mentioned above.

ROUND-ROBIN STUDY

An international round-robin study under the auspices of ISO TC201/SC6 was initiated in July, 2004 to test the issues raised in this paper as a background for preparation of a documentary standard method for depth-profiling of arsenic in silicon. It will also determine the level of interlaboratory agreement that can be achieved for the determination of arsenic implant dose. Three arsenic implants with doses ranging from about $1 \times 10^{13}/\text{cm}^2$ to $2 \times 10^{16}/\text{cm}^2$ were distributed to 16 laboratories with 19 SIMS instruments in 7 countries. Instructions were given to determine their implanted doses using SRM 2134 for calibration. The laboratories were requested to use both Si_2^- and Si_3^- as

reference species with the AsSi^- analyte signal, and to use cycle-by-cycle matrix normalization as well as average matrix signal normalization for the RSF and dose determinations. After the results have been received and tabulated, they will help to determine how the issues raised in this paper will be incorporated into the new documentary standard method.

REFERENCES

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