



Certificate of Analysis

Standard Reference Material® 2134

Arsenic Implant in Silicon Depth Profile Standard

This Standard Reference Material (SRM) is intended for use in calibrating secondary ion response to minor and trace levels of arsenic in a silicon matrix by the analytical technique of secondary ion mass spectrometry (SIMS). SRM 2134 is intended for calibrating the response of a SIMS instrument for arsenic in a silicon matrix under a specific set of instrumental conditions. It may also be used by a laboratory as a transfer standard for the calibration of working standards of arsenic in silicon. A unit of SRM 2134 consists of a 1 cm × 1 cm single crystal silicon substrate that has been ion implanted with the isotope ⁷⁵As at a nominal energy of 100 keV. SRM 2134 is certified for the retained dose of ⁷⁵As atoms. The dose is expressed in units of arsenic mass per unit area. Additional noncertified information about the concentration of arsenic atoms as a function of depth below the surface is provided by SIMS.

Certified Concentration Values: The total retained dose of ⁷⁵As atoms was determined by instrumental neutron activation analysis. Aliquots of two independently prepared arsenic reference solutions (one of which was NIST SRM 3103a Arsenic Standard Solution, certified for arsenic concentration) were deposited on filter paper and served as standards in the analysis. The resulting certified value and expanded uncertainty are

$$\text{Certified Retained Dose of } ^{75}\text{As: } 0.09120 \mu\text{g/cm}^2 \pm 0.00035 \mu\text{g/cm}^2$$

Using a value of 74.9216 g/mol for the isotopic mass of ⁷⁵As, the retained dose is equivalent to

$$7.330 \times 10^{14} \text{ atoms/cm}^2 \pm 0.028 \times 10^{14} \text{ atoms/cm}^2$$

The uncertainty in the certified value is expressed as an expanded uncertainty $U = ku_c$, where k is a coverage factor of 2.04 giving an approximate level of confidence of 95 %, and u_c is the combined standard uncertainty calculated according to the ISO Guide [1]. The combined standard uncertainty is the square root of the sum of squares of the individual standard uncertainties derived or estimated from the measurement process. The Type A and Type B standard uncertainty components arise from measurement uncertainties in the following principal sources: (1) concentrations of comparator standards, (2) neutron fluence exposure differences between samples and standards, (3) measurement replication, (4) counting statistics for standards, (5) pulse pileup, and (6) decay timing effects. All known potential sources of uncertainty have been considered [2-4].

Expiration of Certification: The certification of **SRM 2134** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification were performed by D.S. Simons of the NIST Surface and Microanalysis Science Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
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The ion implantation for this SRM was carried out at Varian Semiconductor Equipment Associates¹ under the direction of D. Downey. Instrumental neutron activation analysis, data reduction, and statistical analysis were performed by R.M. Lindstrom and R.R. Greenberg of the NIST Analytical Chemistry Division. Secondary ion mass spectrometry measurements were performed by P.H. Chi of the NIST Surface and Microanalysis Science Division, and by C.W. Magee and W. Ou of Evans East. Rutherford backscattering measurements were provided by E.D. Adams of IBM Microelectronics. X-ray fluorescence measurements were provided by W.H. Gries of Deutsche Telekom. Cleaning and packaging were performed by C. Ng of the NIST Surface and Microanalysis Science Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Production and Physical Description: The starting material consisted of a commercial 200 mm p-type silicon (100) single crystal wafer polished on one side. The polished side of the silicon wafer was implanted with ⁷⁵As ions at a nominal energy of 100 keV in an ion implanter. The wafer was nominally at room temperature during the implantation. The wafer was cut into 1 cm x 1 cm squares with a wafer saw. All squares were located at least 1 cm from the edge of the wafer.

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Handling: The implanted side of the SRM is the polished, reflective side. This surface was cleaned prior to packaging. Immediately prior to use, dust particles should be removed from the surface with a pressurized air duster.

Etching this SRM in hydrofluoric acid is **NOT** recommended because some arsenic may be removed with the surface oxide.

Storage: When NOT in use the SRM should be stored in its original tray with the cover secured tightly.

Use: Information on material composition as a function of depth can be obtained with SIMS by monitoring one or more sputtered ion species as successive layers are removed by ion bombardment. When using this technique, the concentration value of a species is normally calibrated with a reference sample of the same species in the same matrix as the unknown [5,6].

SUPPLEMENTAL INFORMATION

The usefulness of this SRM for calibrating the SIMS response function of ⁷⁵As in silicon depends on maintaining constant analysis conditions during the acquisition of the SIMS depth profile. Care must be taken to ensure that the primary ion beam current remains stable and the beam raster location remains fixed.

The energy of the ion implanter used in the production of this SRM was not calibrated. Caution should therefore be exercised in comparing the experimental parameters from the SIMS profile with theoretical predictions of implantation models.

The functional form of the concentration versus depth of ⁷⁵As in this material was measured by SIMS under 3 keV Cs⁺ bombardment at an angle of 60° to the surface normal and with ⁷⁵As²⁸Si⁻ detection. SIMS depth-profile data taken with a quadrupole mass spectrometer are plotted in Figure 1. The figure shows that the arsenic concentration decreases as a function of depth over the first 10 nm below the surface. This near-surface component of the arsenic depth distribution is estimated to comprise less than 2 % of the total retained dose. It is included in the certified dose value and should be considered when integrating the SIMS profile to calibrate the SIMS response for arsenic.

The lateral uniformity of the implanted dose was verified by sheet resistance mapping of a companion wafer that was implanted in the same batch as the one used for this SRM. This measurement showed a relative standard deviation of 0.17 % of the mean sheet resistance over 49 analysis points. In addition, the sample-to-sample variation of arsenic content that was observed during the instrumental neutron activation analysis measurements was consistent with the counting statistics combined with other known sources of variation, indicating no significant heterogeneity among the 10 samples measured.

¹ Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Measurements of the implanted arsenic dose by Rutherford backscattering spectrometry and by x-ray fluorescence spectrometry were consistent with the certified dose within their estimated uncertainty intervals.

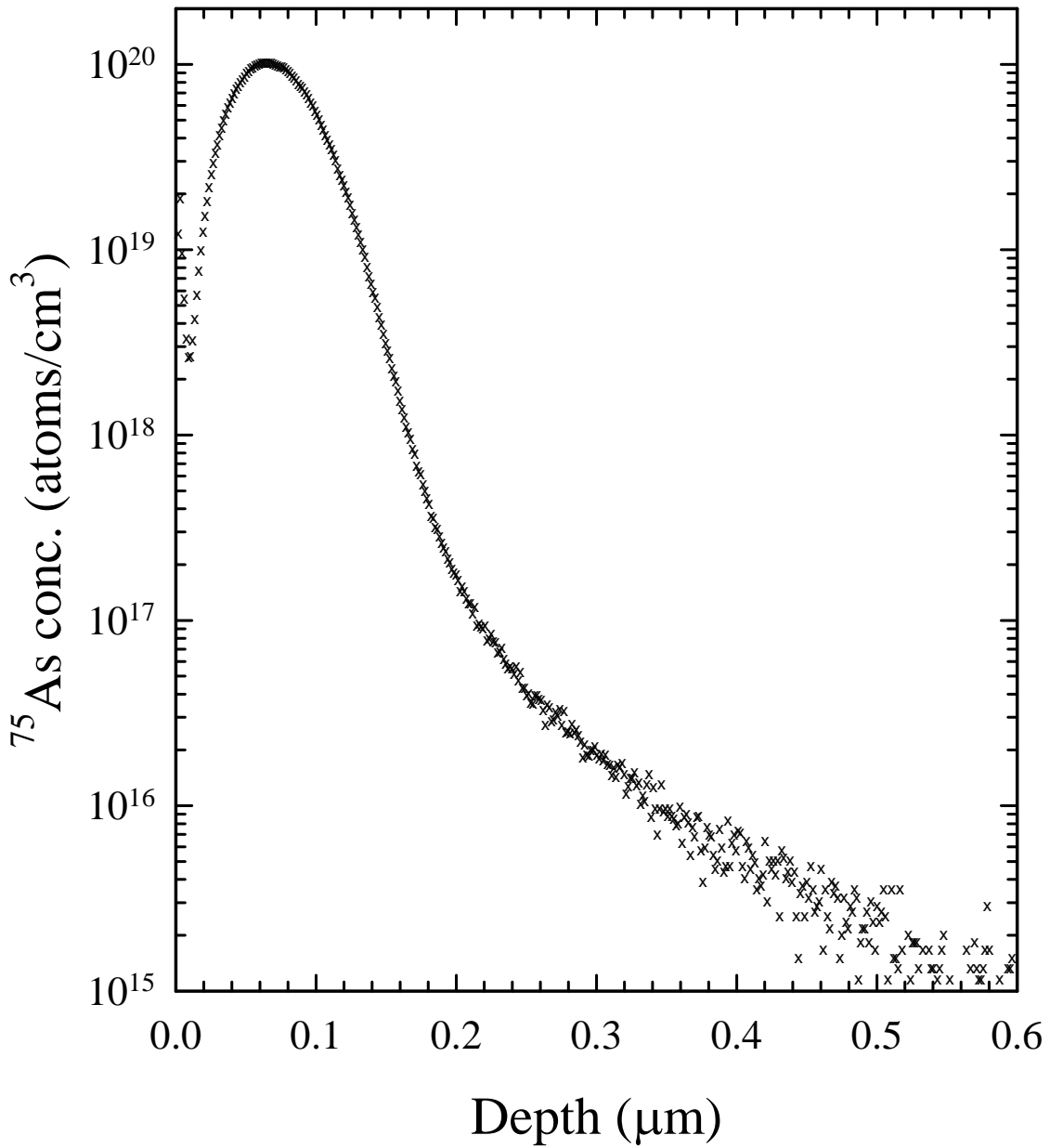


Figure 1. SIMS depth profile of ^{75}As in SRM 2134 using 3 keV Cs^+ ion bombardment at 60° from normal incidence and AsSi^- detection.

REFERENCES

- [1] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2010); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://www.nist.gov/physlab/pubs/index.cfm> (accessed Aug 2010).
- [2] Greenberg, R.R.; Lindstrom, R.M.; Simons, D.S; *Instrumental Neutron Activation Analysis for Certification of Ion-Implanted Arsenic in Silicon*; *Journal of Radioanalytical and Nuclear Chemistry*, Vol. 245, pp. 57–63, (2000).
- [3] Simons, D.S.; Downing, R.G.; Lamaze, G.P.; Lindstrom, R.M.; Greenberg, R.R.; Paul, R.L.; Schiller, S.B; Guthrie, W.F.; *Development of certified reference materials of ion-implanted dopants in silicon for calibration of secondary ion mass spectrometers*; *J. Vac. Sci. Technol. B.*, Vol. 25, pp. 1365–1375 (2007).
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- [5] Wilson, R.G.; Stevie, F.A.; Magee, C.W.; *Secondary Ion Mass Spectrometry - A Practical Handbook for Depth Profiling and Bulk Impurity Analysis*; New York, John Wiley & Sons, Sect. 3.1 (1989).
- [6] ISO 18114:2003; *Surface Chemical Analysis - Secondary Ion Mass Spectrometry – Determination of relative sensitivity factors from ion-implanted reference materials*; International Organization for Standardization, Geneva, Switzerland; available at <http://www.iso.org/iso/home.html> (accessed Aug 2010).

Certificate Revision History: 05 August 2010 (Extension of the certification period; addition of journal references, editorial changes); 09 August 2000 (Original certification date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975 2200; fax (301) 926 4751; e mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.