National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2133

Phosphorus 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 phosphorus in a silicon matrix by the analytical technique of secondary ion mass spectrometry (SIMS). SRM 2133 is intended for calibrating the response of a SIMS instrument for phosphorus 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 phosphorus in silicon. This SRM consists of a 1 cm × 1 cm single crystal silicon substrate that has been ion-implanted with the isotope ^{31}P at a nominal energy of 100 keV.

SRM 2133 is certified for the retained dose of ^{31}P atoms. The dose is expressed in units of phosphorus mass per unit area. Additional non-certified information about the concentration of phosphorus atoms as a function of depth below the surface is provided based on SIMS analysis.

Certified Value and Uncertainty: The total retained dose of ^{31}P atoms was determined by radiochemical neutron activation analysis (RNAA). Aliquots of two independently prepared phosphorus reference solutions (one of which was NIST SRM 3139a *Phosphorus Standard Solution*, certified for phosphorus concentration) were deposited on aluminum foils and served as standards. The resulting certified value and expanded uncertainty are:

Certified Retained dose of ^{31}P: $0.04927\ \mu g/cm^2 \pm 0.00083\ \mu g/cm^2$

Using a value of 30.97376 g/mol for the isotopic mass of ^{31}P, the retained dose is equivalent to:

$$9.58 \times 10^{14}\ atoms/cm^2 \pm 0.16 \times 10^{14}\ atoms/cm^2$$

The uncertainty in the certified value is expressed as an expanded uncertainty $U = ku_c$, where k is a coverage factor of 2.0 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 obtained by combining the individual standard uncertainties derived or estimated from the measurement process by propagation of uncertainties [1]. The Type A and Type B standard uncertainty components arise from measurement uncertainties in the following principal sources: (1) measurement replication and heterogeneity for samples, (2) measurement replication for comparator standards, (3) concentrations of standard solutions, (4) radiochemical impurities, (5) volumetric calibrations of standards, and (6) determination of carrier yield. All known potential sources of uncertainty have been considered [2-4].

Expiration of Certification: The certification of **SRM 2133** 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.

John A. Small, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 05 August 2010
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Robert L. Watters, Jr., Chief
Measurement Services Division

Statistical analysis was performed by W.F. Guthrie and J. Lu of the NIST Statistical Engineering Division and R.L. Paul, with assistance from R.R. Greenberg of the NIST Analytical Chemistry Division.

The ion implantation for this SRM was carried out under the direction of L. Rubin of Axcelis Technologies[1].

Radiochemical neutron activation analysis (RNAA) and data reduction were performed by R.L. Paul of the NIST Analytical Chemistry Division.

Secondary ion mass spectrometry measurements were performed by P.H. Chi and D.S. Simons of the NIST Surface and Microanalysis Science Division and by C.W. Magee of Evans East.

Cleaning and packaging were performed by D.S. Simons 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 ^{31}P 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 × 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 duster.

Etching the sample in hydrofluoric acid is **NOT** recommended because some phosphorus 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. 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 ^{31}P 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 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 ^{31}P in this material was measured by SIMS under 3 keV Cs^+ bombardment at an angle of 60° to the surface normal and with $^{31}P^-$ detection. SIMS depth-profile data taken with a quadrupole mass spectrometer are plotted in Figure 1.

The lateral variation of the implanted dose was tested 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 0.43 % of the mean sheet resistance over 81 analysis points. The sample-to-sample variation of phosphorus content that was observed during the neutron activation analysis measurements was modeled as a function of sample location. The uncertainty in the certified value takes into account the observed heterogeneity among samples.

[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.

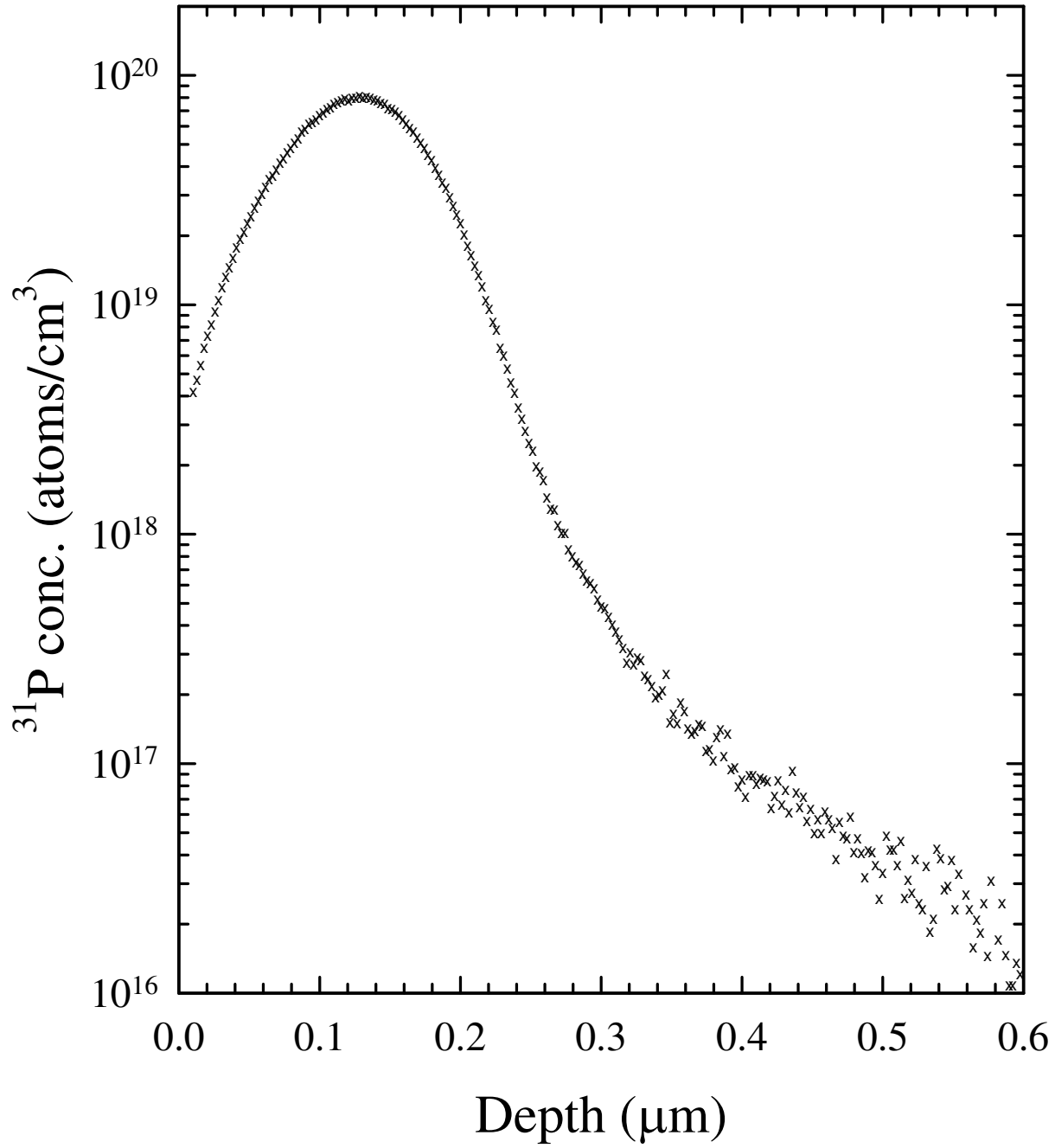

Figure 1. SIMS depth profile of ^{31}P in SRM 2133 using 3 keV Cs^+ ion bombardment at 60° from normal incidence and $^{31}P^-$ detection.

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/utils/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] Paul, R.L.; Simons, D.S.; Guthrie, W.F.; Lu, J.; *Radiochemical Neutron Activation Analysis for Certification of Ion-Implanted Phosphorus in Silicon*; Anal. Chem., Vol. 75, pp. 4028-4033 (2003).

[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).

[4] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed Aug 2010).

[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); **01 July 2004** (Editorial change to update reference #2); **02 April 2003** (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.