National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2137

Boron Implant in Silicon Standard for Calibration of Concentration in a Depth Profile

Serial No: SAMPLE

This Standard Reference Material (SRM) is intended for use in calibrating the secondary ion response to minor and trace levels of boron in a silicon matrix by the analytical technique of secondary ion mass spectrometry (SIMS). A unit of SRM 2137 consists of a single crystal silicon substrate with a surface rendered disordered by silicon ion implantation. The substrate is ion-implanted with the isotope ^{10}B at a nominal energy of 50 keV. SRM 2137 is certified for the retained dose of ^{10}B atoms by neutron depth profiling. The dose is expressed in units of ^{10}B mass per unit area. Noncertified information about the concentration of ^{10}B atoms as a function of depth below the surface is provided by SIMS.

Certified Value and Uncertainty: The total retained dose of ^{10}B atoms was determined by a neutron reaction method known as neutron depth profiling, using a calibrated reference film of metallic ^{10}B deposited on silicon as a standard [1].

Retained dose of ^{10}B		Total Uncertainty
0.01692 μg/cm^2	±	0.00059 μg/cm^2

Using a value of 10.012937 g/mol for the isotopic mass of ^{10}B, the retained dose is equivalent to:

$$1.018 \times 10^{15} \text{ atoms/cm}^2 \quad \pm \quad 0.035 \times 10^{15} \text{ atoms/cm}^2$$

The stated uncertainty is a 95 % coverage, 95 % confidence tolerance interval that includes random measurement error, estimates of material variability, and systematic uncertainties. The assumption has been made that the actual retained dose may vary among SRM units due to spatial variation in the implantation process. The interval defined by the certified value and its associated uncertainty covers the true ^{10}B dose in at least 95 % of the SRM units, with 95 % confidence.

Expiration of Certification: The certification of **SRM 2137** 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.

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 S.B. Schiller and L.M. Oakley of the NIST Statistical Engineering Division.

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Certificate Issue Date: 05 August 2010
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
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Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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.

Physical Description: The production of the SRM involved several separate steps. The starting materials were commercial n-type silicon (100) single crystal wafers of 76-mm diameter, polished on one side. A grid pattern of 5-mm squares was made on the unpolished side of the wafer by a photolithographic method. Each 5-mm square contains row, column, and wafer numbers that provide unique locational identification.

The polished side of the silicon wafer was implanted with ^{28}Si ions in an ion implanter to render the surface sufficiently disordered that it would appear to be amorphous to the ^{10}B ion beam. The ^{28}Si ions were implanted in 3 stages: 2×10^{15} atoms/cm^2 at 360 keV (as Si^{++}); 2×10^{15} atoms/cm^2 at 140 keV (as Si^{+}); and 2×10^{15} atoms/cm^2 at 50 keV (as Si^{+}). Then ^{10}B was implanted at a nominal energy of 50 keV and a nominal dose of 1×10^{15} atoms/cm^2. The wafers were nominally at room temperature during all implantations.

The wafers were cut into 1 × 1 cm squares with a wafer saw. All squares were located at least 7 mm from the edge of a wafer. The implanted face is the polished, reflective surface.

The ion implantation for this SRM was carried out at the NIST Semiconductor Electronics Division by D.B. Novotny and R.G. Russell, and at the Naval Research Laboratory by H.B. Dietrich and F. Moore. The grid patterning was done by M.L. Miller and wafer dicing was done by J.E. Luther and M.L. Miller of the NIST Semiconductor Electronics Division. Neutron depth profiling measurements were performed by R.G. Downing and G.P. Lamaze of the NIST Inorganic Analytical Research Division. Secondary ion mass spectrometry measurements were performed by D.S. Simons and P.H. Chi of the NIST Surface and Microanalysis Science Division, and by C.W. Magee and S.W. Novak of Evans East, Inc. Sheet resistance mapping measurements for wafer homogeneity were performed by J.R. Ehrstein of the NIST Semiconductor Electronics Division.

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 HF is **NOT** recommended, because some boron may be removed with the surface oxide, and also because the grid pattern on the back will be removed.

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 [2,3]. SRM 2137 is intended for calibrating the response of a SIMS instrument for ^{10}B 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 ^{10}B in silicon. The determination of the response of a SIMS instrument for ^{11}B requires additional knowledge about mass bias effects in SIMS for boron isotopes. The relative response function of the SIMS instrument for ^{11}B with respect to ^{10}B in a silicon matrix can be measured with the aid of a specimen of silicon that contains a bulk doping of elemental boron with a natural isotopic composition (80.1 atom percent ^{11}B, 19.9 atom percent ^{10}B).

SUPPLEMENTAL INFORMATION

The usefulness of this SRM for calibrating the SIMS response function of ^{10}B in silicon depends on maintaining constant analysis conditions during the acquisition of the SIMS 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 user of this SRM should be aware of a possible mass spectral interference between ^{10}B and $^{30}Si^{3+}$ that may be significant under certain SIMS measurement conditions.

The functional form of the concentration vs depth of ^{10}B in this material was measured by SIMS under conditions that minimize ion-induced mixing effects (3 keV O_2^+ bombardment at an angle of 52° to the surface normal). SIMS depth-profile data from eight profiles from two areas on each of two different wafers are plotted together in Figure 1. Data points within 15 nm of the surface are not indicative of the true surface concentration of ^{10}B due to the presence of a native oxide on the silicon and transient effects that occur following the initiation of ion sputtering. Concentration values below 10^{16} atoms/cm^3 of ^{10}B may not be indicative of the true depth distribution of ^{10}B because of background signals in the SIMS measurement.

The maximum ^{10}B concentration of 8.37×10^{19} atoms/cm^3 occurs at a depth of 0.188 μm. Interpolated and averaged SIMS data from Figure 1, excluding values for depths less than 20 nm from the surface and concentrations below 10^{16} atoms/cm^3, are provided in Table 1 for reference.

The total ^{11}B content within 0.5 μm of the surface is less than 10^{13} atoms/cm^2, as determined by SIMS.

Table 1. Noncertified SIMS Values of Concentration of ^{10}B vs Depth for SRM 2137

Depth (μm)	^{10}B Conc. (atoms/cm^3)	Depth (μm)	^{10}B Conc. (atoms/cm^3)
0.020	3.90×10^{18}	0.175	8.05×10^{19}
0.025	4.50×10^{18}	0.180	8.23×10^{19}
0.030	5.17×10^{18}	0.185	8.34×10^{19}
0.035	5.86×10^{18}	0.190	8.36×10^{19}
0.040	6.63×10^{18}	0.195	8.27×10^{19}
0.045	7.53×10^{18}	0.200	8.11×10^{19}
0.050	8.49×10^{18}	0.205	7.83×10^{19}
0.055	9.59×10^{18}	0.210	7.43×10^{19}
0.060	1.08×10^{19}	0.215	6.93×10^{19}
0.065	1.22×10^{19}	0.220	6.33×10^{19}
0.070	1.36×10^{19}	0.225	5.68×10^{19}
0.075	1.54×10^{19}	0.230	4.98×10^{19}
0.080	1.71×10^{19}	0.235	4.28×10^{19}
0.085	1.91×10^{19}	0.240	3.57×10^{19}
0.090	2.14×10^{19}	0.245	2.91×10^{19}
0.095	2.38×10^{19}	0.250	2.29×10^{19}
0.100	2.65×10^{19}	0.255	1.76×10^{19}
0.105	2.93×10^{19}	0.260	1.30×10^{19}
0.110	3.24×10^{19}	0.265	9.25×10^{18}
0.115	3.56×10^{19}	0.270	6.37×10^{18}
0.120	3.90×10^{19}	0.275	4.20×10^{18}
0.125	4.28×10^{19}	0.280	2.65×10^{18}
0.130	4.67×10^{19}	0.285	1.61×10^{18}
0.135	5.08×10^{19}	0.290	9.25×10^{17}
0.140	5.49×10^{19}	0.295	5.01×10^{17}
0.145	5.90×10^{19}	0.300	2.70×10^{17}
0.150	6.32×10^{19}	0.305	1.34×10^{17}
0.155	6.72×10^{19}	0.310	6.87×10^{16}
0.160	7.11×10^{19}	0.315	3.26×10^{16}
0.165	7.48×10^{19}	0.320	1.69×10^{16}
0.170	7.79×10^{19}		

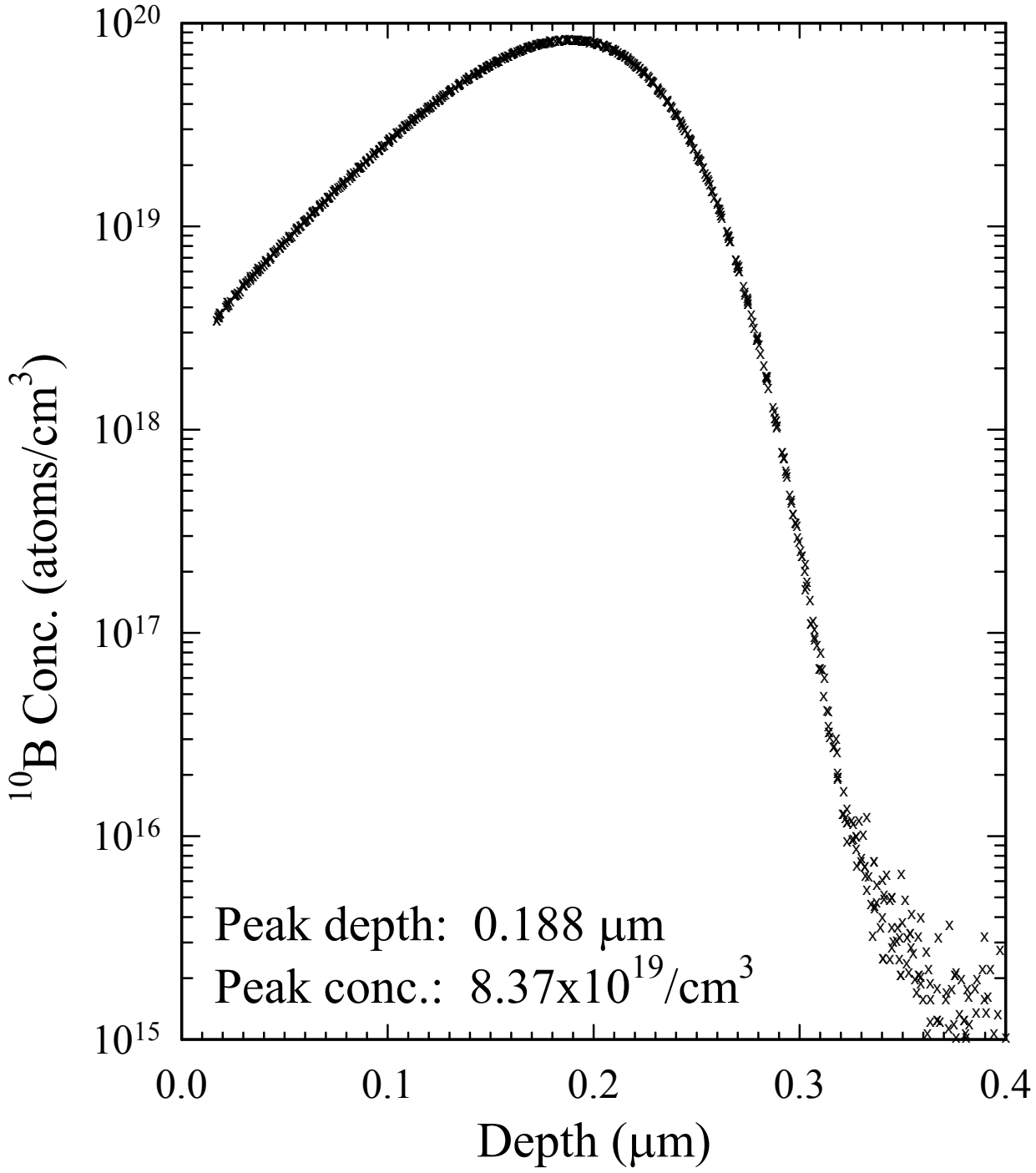

Figure 1. Eight superimposed SIMS depth profiles of ^{10}B in SRM 2137 using 3 keV O_2^+ ion bombardment at 52° from normal.

REFERENCES

[1] 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).

[2] 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).

[3] 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 certification period; addition of a journal reference, editorial changes); **13 August 1993** (Original certificate 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.