



Designation: F 2139 – 01

Standard Test Method for Measuring Nitrogen Concentration in Silicon Substrates by Secondary Ion Mass Spectrometry¹

This standard is issued under the fixed designation F 2139; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of total nitrogen concentration in the bulk of single crystal substrates using secondary ion mass spectrometry (SIMS) (1,2)².

1.2 This test method can be used for silicon in which the dopant concentrations are less than 0.2 % (1×10^{20} atoms/cm³) for boron, antimony, arsenic, and phosphorus.

1.3 This test method is for bulk analysis where the nitrogen concentration is constant with depth.

1.4 This test method can be used for silicon in which the nitrogen content is 1×10^{14} atoms/cm³ or greater. The detection capability depends upon the SIMS instrumental nitrogen background and the precision of the measurement.

1.5 This test method is complementary to infrared spectroscopy, electron paramagnetic resonance, deep level transient spectroscopy, and charged particle activation analysis (3). The infrared spectroscopy method detects nitrogen in specific vibrational states, rather than total nitrogen, and is limited to silicon with doping concentrations less than about 1×10^{17} atoms/cm³. The charged particle activation analysis detection capability is limited by an interference from boron.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process³
- E 673 Terminology Relating to Surface Analysis⁴

3. Terminology

3.1 **Definitions**—All terms in this test method are in accordance with those given in Terminology E 673.

4. Summary of Test Method

4.1 SIMS is utilized to determine the bulk concentration of nitrogen in single crystal silicon substrate. Specimens of single crystal silicon (one calibration specimen and the test specimens) are loaded into a sample holder. The holder with the specimens is baked at 100°C in air for 1 h and then transferred into the analysis chamber of the SIMS instrument.

4.2 The calibration sample is analyzed using a cesium (Cs) primary ion beam and negative ion spectrometry of ¹⁴N²⁸Si, or ¹⁵N²⁸Si, depending upon the nitrogen isotope in the calibration sample, to determine a relative sensitivity factor (RSF) of nitrogen in silicon.

4.3 Each test specimen in the sample holder is sputtered by the primary cesium ion beam, without analyzing secondary ion intensities, in order to reduce the nitrogen background. The amount of time for this pre-analysis sputtering will depend upon the instrumentation and the desired background nitrogen concentration.

4.4 For the analysis of the test specimens, a cesium primary ion beam is used to bombard each test specimen at two different sputter rates, by reducing the beam raster area for the second sputter rate, while at the same time maintaining a constant detected area. This beam raster change technique is used for two reasons. First, this technique reduces the background nitrogen contribution from the sample surface. Secondly, this technique allows the determination of both the instrumental nitrogen background and the real bulk nitrogen concentration in each test specimen measurement without the use of a separate blank specimen, and due to averaging is capable of separating the bulk nitrogen from the instrumental nitrogen even if the bulk nitrogen is less than the instrumental nitrogen (4).

4.5 The two sputter rates, and the amount of time they are applied in each measurement, are chosen to optimize the detection capability, and will vary with instrumentation. However, the second sputter rate is typically the maximum sputter rate capable of the instrumentation, and the first sputter rate is

¹ This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Oct. 10, 2001. Published December 2001.

² The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.



typically less than half that of the second sputter rate.

4.6 The negative secondary ion of $^{14}\text{N}^{28}\text{Si}$ is mass analyzed by a mass spectrometer, and detected by an electron multiplier (EM) or equivalent high-sensitivity ion detector as a function of time. The matrix negative secondary ion count for silicon (^{28}Si , ^{29}Si , ^{30}Si , or a dimer such as $^{30}\text{Si}_2$) is measured by a faraday cup (FC) or appropriate detector during the profile. If multiple detectors are used during the test, the relative sensitivities of the detectors are determined by measuring standard ion signals (either the same negative secondary ion count rate or ion count rates of known relative intensity such as natural $^{28}\text{Si}/^{30}\text{Si}$) on each detector. Additional negative secondary ions of $^{11}\text{B}^{28}\text{Si}$, ^{12}C , and $^{12}\text{C}^{28}\text{Si}$ may also be detected depending upon the boron and carbon concentration in the test specimen.

4.7 For illustration purposes an example data profile is shown in Fig. 1 where the log of counts of $^{12}\text{C}^{28}\text{Si}$, $^{14}\text{N}^{28}\text{Si}$, and ^{30}Si is plotted versus sputter time. At about 200 s, the Cs beam raster area was changed from $250\text{ }\mu\text{m} \times 250\text{ }\mu\text{m}$ down to $50\text{ }\mu\text{m} \times 50\text{ }\mu\text{m}$, resulting in an increase in signal intensities, because the volume of material analyzed per second has increased. At about 350 s, the Cs beam raster area was reversed back to the first condition, and the signal intensities are seen to be reduced back to the original levels. Since the surface analysis area was kept constant during the raster change (that is, the actual surface area detected is independent of the raster area), the signal intensities have two separate contributions, one from elements that are adsorbed to the surface and one from elements in the volume of material analyzed each second. There is some nitrogen (and carbon in this case) that is adsorbed to the surface from the local environment outside of

the silicon, and there is some nitrogen that is from the bulk silicon. A key point is that if there is no nitrogen in the bulk silicon, the nitrogen signal intensity will not change as a result of the change in sputter rate, because the adsorbed nitrogen (that is, instrumental nitrogen signal) is only dependent on the analysis area, which has been held constant. This understanding leads to the following equations:

$$I_n/I_{si} \times RSF = [N_b] + [N]$$

$$I_N/I_{SI} \times RSF = [N_B] + [N]$$

$$[N_b] = I_B/I_{si} \times RSF$$

$$[N_B] = I_B/I_{SI} \times RSF$$

where I_n and I_N are the secondary ion intensities of $^{14}\text{N}^{28}\text{Si}$ under two sputter rate conditions, I_n corresponding to the higher sputter rate and I_N corresponding to the slower sputter rate; $[N_b]$ and $[N_B]$ are the contributions of background nitrogen to the observed nitrogen concentration under the conditions of the two sputter rates, $[N_b]$ corresponding to the higher sputter rate; I_B is the secondary ion intensity from the adsorbed background nitrogen under the conditions of both sputter rates; I_{si} and I_{SI} are the secondary ion intensities of the silicon matrix, ^{30}Si in our example, under the two sputter rates, I_{si} corresponding to the higher sputter rate; and RSF is a relative sensitivity factor for converting the ion intensity ratio to concentration; and $[N]$ is the bulk nitrogen concentration in the test specimen.

4.8 The equations in 4.7 can be used to determine $[N]$ and $[N_b]$ from measured data as follows:

$$[N] = RSF \times \{I_n/I_{si}\} \times \{1 - I_N/I_n\} / \{1 - I_{SI}/I_{si}\}$$

$$[N_b] = RSF \times \{I_n/I_{si}\} - [N]$$

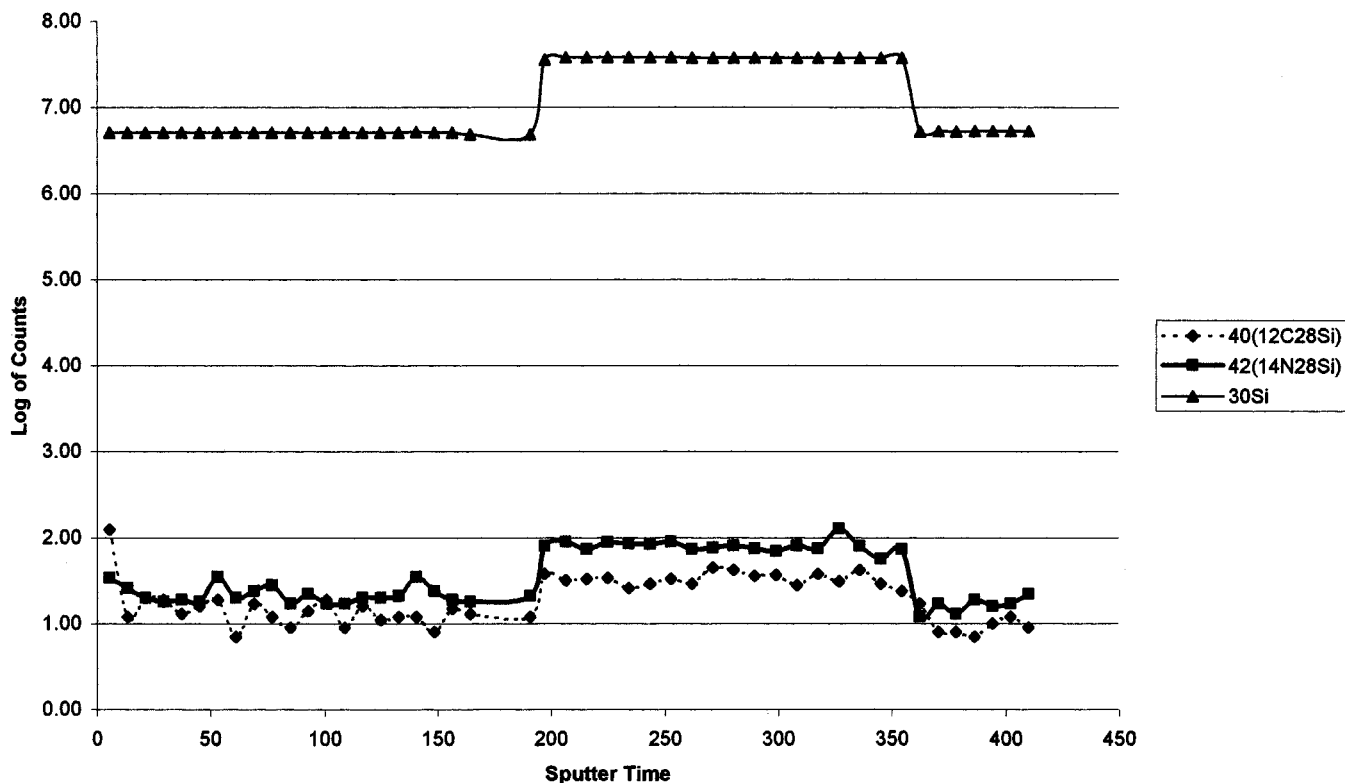


FIG. 1 SIMS Signals in Nitrogen-Doped Silicon, Log Scale