**A method for quantitative nanoscale imaging of dopant distributions using secondary ion mass spectrometry: an application example in silicon photovoltaics**

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(Received 23 January 2019; accepted 14 June 2019)

**Abstract**

A method for rapid quantitative imaging of dopant distribution using secondary ion mass spectrometry (SIMS) is described. The method is based on SIMS imaging of the cross-section of a reference sample with a known concentration profile. It is demonstrated for the case of boron quantification in silicon in a SIMS imaging mode. A nonlinear relationship between the secondary ion intensity and the concentration is observed. A detection limit of $3 \pm 2 \times 10^{17} \text{at./cm}^2$ ($\sim 6$ ppm) is determined with 39 nm pixel-size for the used experimental conditions. As an application example, a boron concentration profile in a passivating contact deposited on a textured Si surface is analyzed.

**Introduction**

Quantitative nanoscale chemical imaging of elements present in low concentrations is required in many areas of scientific research. The chemical, physical, and electronic properties of materials can be dramatically changed by additions of even small concentrations of doping or alloying elements. For example, dopants are used in semiconductor materials to tune their electronic properties. Likewise, small additions of carbon (C) or boron (B) in steel are known to drastically change their mechanical properties. Modern materials are often designed with low concentrations of elements with complex nanoscale structural features to improve their performance. To establish the link between macroscale properties and the local chemical characteristics, quantitative nanoscale mapping of trace concentrations of elements is essential.

Secondary ion mass spectrometry (SIMS) is a well-known high-sensitivity chemical analysis technique widely used for the investigation of chemical composition even down to ppm levels of concentration. However, the quantification of SIMS intensities is not straightforward mainly because of the strong variations in ionization yield for a given element as a function of the matrix elements (i.e., matrix effect). Another limitation of SIMS is the lateral resolution which is fundamentally limited by the ion–solid interaction volume size of $\sim 10$ nm. To overcome this limitation, a correlative microscopy approach is often employed. In this context, it should be noted that atom probe tomography (APT) can provide quantitative high-resolution and high-sensitivity imaging of the elements, but the main limitations of this technique are that the sample preparation is very laborious and, more importantly, the analysis volume is extremely small ($\sim 100$ nm needles with tip radius of $<10$ nm and volume in the $10^{-8}$ nm$^3$ range). Hence, in contrast to SIMS, the analysis of fields of view (FOV) in the micrometer length scales is not possible with the APT technique.

A well-known method often used in the quantification of SIMS depth profiles (i.e., 1D analysis) is by using reference samples of known concentrations obtained by ion implantation.
or by a dedicated preparation of special sets of samples containing
the known concentration of elements distributed uniformly.

The latter method has been successfully used in the past also to
quantify SIMS images\cite{6,9} (i.e., 2D analysis) in multiphase
steels. Carbon contents were determined from a calibration
curve established from dual-phase steels with known concen-
trations of carbon. The main limitation of that approach is
that dedicated sets of special reference samples are needed to
obtain the calibration curve and only few discrete points in
the concentration space are covered.

In the present work, we introduce a new method for direct
quantitative SIMS imaging based on cross-sectional imaging
of ion-implanted reference samples prepared using optimal
ion dose and implantation energy. The main advantage of
this new approach is that within a single image acquisition,
reference concentrations varying continuously over few orders of
magnitude can be determined. This conversion can then be
applied to other samples with the same matrix as the reference
sample. We demonstrate this method with an application exam-
ples in the analysis of dopant concentrations in carrier-selective
passivating contacts used in c-Si solar cells.\cite{10,11} A method
commonly used in the solar cell industry to assess dopant pro-
files is electrochemical capacitance voltage profiling. However,
this technique reflects only the active dopant concentration and
not their total concentration, and since it is a 1D method, it does
not reveal local variations. We apply the new method for quant-
itative nanoscale SIMS imaging to the laterally inhomoge-
neous dopant distribution across the deposited layers and the
in-diffused region of a passivating contact in a photovoltaic
material with surface texture in order to better understand its
macroscopic electronic properties. The method can also be
broadly applied to other areas of research.

**Experimental methods**

**Reference samples**

A 4″ silicon (Si) wafer with a crystal orientation (100) was
implanted with B with implantation energy of 190 keV and a
dose of $10^{16}$ ions/cm$^2$ at the Surrey Ion Beam Centre,
Guildford, UK. The specified dose accuracy from the measure-
ments is better than 1% in both uniformity and the absolute
value. The implantation was performed over the full 4″
wafer, apart from an approximate exclusion zone of around 3
mm all around the edge of the wafer where it is held in place
during the irradiation. The irradiation took place at 7° off
axis with a 22° twist to avoid channeling down the (100) direc-
tion. The energy of implantation was chosen such that the con-
centration maximum forms as deep as possible from the surface
and to have an extended concentration profile. This is necessary
to have a sufficient number of data points (i.e., pixels) across
the concentration profile during the subsequent cross-sectional
SIMS imaging. A Monte Carlo-based simulation program,
SRIM (Version 2013),\cite{12} was used to estimate the concentra-
tion profile as a function of the depth. The maximum concen-
tration of B according to the simulation is $5 \times 10^{20}$ at./cm$^3$ (or
1 at.%) at a depth of 550 nm from the surface. In addition,
SDTRIMSP code\cite{13} was used to simulate B implantation
into Si. Further details of the simulations are provided in
Supplementary Material.

**Passivating contact samples for solar cells**

The test structures for passivating solar cell contacts were fab-
ricated on symmetrically processed p-type float zone Si wafers
with [100] crystal orientation. After KOH texturing and clean-
ing, a thin (~1 nm) $\text{SiO}_2$ chemical oxide was grown by wet
chemical oxidation of the substrate in hot nitric acid.

Subsequently, a 30-nm thick layer of boron-doped $\text{SiCx-Hx}$
was deposited by parallel-plate plasma-enhanced chemical
vapour deposition. The deposition was done on both sides
such that the layers are symmetrically deposited on the Si sub-
strate. The boron diffusion was then obtained by annealing the
sample at 850 °C in a quartz tube furnace under inert gas atmos-
phere without dwell time at peak temperature. Thereafter, the
sample was covered on both sides with a $\text{SiN}_x$ layer which
serves as a hydrogen donor. Hydrogen is released from this
layer by annealing at 450 °C on a hot plate. Eventually, the
$\text{SiN}_x$ layer was removed in hydrofluoric acid (HF). The process
is described elsewhere in detail.\cite{10,14} A schematic of the final
stacking of the layers on the textured Si is shown in the
Results section (Fig. 2(a)).

For SIMS 2D imaging, the cross-section of the ion-
implanted reference sample as well as of the textured solar
cell sample was prepared as follows. First, the samples were
cleaved and then the cross-sections were exposed by sandwich-
ing two pieces of the same type face-to-face using an epoxy
glue. After curing for 24 h, the entire sandwich assemblies
were embedded into larger epoxy discs. The embedded cross-
sectional samples were then mechanically polished to mirror
finish. For transmission electron microscopy (TEM), the cross-
sectional sample of the textured solar cell sample was prepared
by conventional focused ion beam (FIB) preparation in the
form of H-bar.\cite{15}

SIMS analyses were performed in 1D (i.e., depth profiling)
mode as well as 2D (i.e., imaging) mode. SIMS depth profiling
was done using a Cameca SC-Ultra with $\text{O}_2^+$ primary ions with a
primary energy and current of 7 keV and 85 nA, respectively.
For these conditions, the SIMS depth resolution is estimated\cite{16}
to be ~10 nm and an estimated slope of ~20 nm per decade.
The depth was calibrated by measuring the crater depth using a
KLA-Tencor P17 profilometer. For SIMS imaging, a
Cameca NanoSIMMS50 instrument with an $\text{O}^+$ primary ion
beam with an impact energy of 16 keV was used. The probe
current was 12 pA and the probe size was estimated to be
400 nm. The SIMS images of $^{11}\text{B}$, $^{12}\text{C}$, and $^{28}\text{Si}$
were obtained simultaneously in matrices of 256 × 256 pixels. The
dwell time was 10 and 20 ms per pixel for FOV of 10 × 10
and 40 × 40 μm$^2$, respectively. Mass resolving power was set
to 3000. The cross-sectional reference sample and the solar
cell sample were imaged consecutively at exactly identical
conditions. Before the acquisition of SIMS images, the area of
interest is irradiated with primary ions to clean the surface.
This is a standard procedure to remove possible surface modification due to exposure to air during sample transfer. Furthermore, as the primary ions were O\(^-\), a steady-state oxygen concentration in the matrix is reached resulting in enhanced ionization yields.

An FEI Tecnai G2 F20 TEM operating at 200 keV was used for imaging in the scanning TEM (STEM) mode, and the high-angle annular dark field (HAADF) images were recorded using a Fischione Model 3000 HAADF detector. Scanning electron microscopic (SEM) images were acquired in a secondary electron (SE) mode in an FEI Scios operating with a 10 keV primary electron energy.

**Results and discussion**

The central idea of the SIMS image quantification method presented here is based on the comparison of intensity profile across a sample of known concentration profile with the intensities in a sample of unknown concentration. Figure 1(a) shows a cross-sectional SIMS image from the ion-implanted reference sample. Figure 1(b) shows the B-depth profile (in blue) in Si quantified using the ion implantation parameters described earlier. The quantity of boron implanted in silicon is known (10\(^{16}\) at./cm\(^2\)), and it corresponds to the integrated signal below the curve B/Si over the depth of implantation. This is the common method used to correlate B intensity to concentration.\(^{16}\)

An intensity profile (in red) measured from the SIMS cross-section image is superposed together with the depth profile in Fig. 1(b). The results indicate a maximum B concentration of 4.8 \times 10^{20} at./cm\(^3\) at a depth of \(~500\) nm. The direction of the intensity profile as well as the integration width are indicated in Fig. 1(a). For comparison, the simulated implantation profile is also shown in Fig. 1(b), and a good agreement between experimental and simulated data is observed. The maximum value of concentration determined from simulation (\(~5 \times 10^{20}\) at./cm\(^3\)) agrees well with the experimentally determined concentration. There is, however, a minor offset in the peak positions of \(~100\) nm. This discrepancy could be attributed to both computational (e.g., parameters used for SDTRIMSP which was set to reproduce TRIM, i.e. *Transport of ions in matter* data) and experimental (e.g., calibration) errors. On the leading edge, the experimental depth profile and the simulated implantation profile agree well. The difference in the trailing edge between experimental depth profile and simulated data is at least partially due to the atomic intermixing and the possible presence of irradiation-induced diffusion during depth profiling with the O\(^+\) beam. In comparison, the simulated data show only the implantation profile and does not capture these processes which occur during depth profiling.

With the help of the superposed curves in Fig. 1(b), the \(^{11}\)B\(^+\) intensity can be calibrated to B concentration as shown in the
inset. Doing so, a nonlinear correlation between the intensity and the concentration is observed within a single sample. The standard method of the quantification of SIMS depth profiles assumes a linear approximation for the relationship between the intensity and the concentration. Interestingly, when we correlate the NanoSIMS line profiles and the concentration, the relationship is found to be nonlinear. We do not believe that this is linked to the averaging effect of the probe size as that would only smoothen rather than accentuate a nonlinear relationship. The results suggest a nonlinear relationship between the intensity and the concentration in the investigated concentration range. From the plots shown in Fig. 1(b), we also note that the line scan intensity from the SIMS image reaches 1 count per voxel (at \( x = 1.25 \mu m \)) and the concentration at that point \( \sim 3 (\pm 2) \times 10^{17} \) at./cm\(^3\), which quantifies the detection limit (defined here as the concentration corresponding to an average intensity of 1 count per voxel) for a pixel-size of 39 nm and the applied SIMS imaging conditions. Further considerations in the determination of detection limit are discussed in Supplementary Material.

It is worth noting that the intensity profile will be a convolution of the actual concentration profile \( C \) (ignoring matrix effect, etc.) and the Gaussian probe profile \( P \). Hence, intensity \( I = C \ast P \). Mathematically, a deconvolution of the probe profile from the experimental line profile would be expected to “sharpen” the profile by eliminating Gaussian broadening. In practice, however, it works only when the input profiles are smooth such that a finite Fourier series expansion is sufficiently accurate, and more importantly, the high-frequency noise background gets amplified during deconvolution. For a robust and reliable deconvolution, the actual beam profile characteristics (such as Gaussian vs. Lorentzian and optical aberrations) will be needed, and the experimental data should not be noisy. Experimentally, a smaller probe size and a wider B-rich zone (e.g., higher implantation energies) would help to mitigate the effects of probe size on the intensity profiles.

SIMS quantification in depth profiling mode is often done by taking the ratio of the intensity of minor element to major element such as B/Si. This normalization step is done to eliminate variations in B intensity due to factors other than real variation in concentration. This approach has the limitation that intensities should be sufficiently high (>10\(^3\) counts per second) to suppress Poisson noise, and it also assumes that the concentration of the major element is essentially invariant. The normalization of B/Si is a standard approach in depth profiling wherein the signal is averaged over the whole FOV. The
same approach cannot be directly applied for quantitative SIMS imaging, the main reason being that a SIMS image may contain two or more types of materials with completely different chemical compositions within the FOV. When a material, which does not contain Si, is present together with another material containing Si, the division of B by Si will result in division by 0 resulting in completely meaningless pixel values. Likewise, Si may not be a major element across the full FOV of the SIMS image. In such cases, the division of B by Si will introduce artifacts. Furthermore, the noise in Poisson statistics is given by \( \sqrt{N} \), where \( N \) is the pixel intensity. Thus, the signal intensities of dopants are noisier in an imaging mode than in the case of depth profiling. Hence, small fluctuations in the intensity will be artificially amplified by taking the B/Si normalization method. For these reasons, we do not apply this method to quantify SIMS images. To improve the signal-to-noise ratio in individual pixels, in the present approach, the line profile in Fig. 1(b) is integrated over the full image width of 256 pixels. An even level of Si intensity is taken as an indication of the absence of spurious fluctuations in primary ion current and other parameters affecting \( ^{11}\text{B}^+ \) signal intensity. Thus, the \( ^{11}\text{B}^+ \) intensity is directly calibrated to the B concentration as shown in the inset of Fig. 1(b).

In this context, it should be noted that there is a well-known inverse relationship between resolution and the physically possible lowest measurable concentration.\(^{[5]}\) Briefly, when the voxel gets smaller (i.e., higher resolution), the number of atoms it can physically contain also get smaller thereby limiting the lowest theoretically detectable concentration. This fundamental link between resolution and the lowest concentration sets a physical limit. The results shown in Fig. 1(a) also allow us to compare the experimental detection limit with the theoretical limit based on the voxel dimensions of the SIMS image. The individual pixel size in the SIMS image shown in Fig. 1(a) is 39 nm, and the image was obtained by summing four planes of images.

The primary current was 12 pA, the probe size was \(~400\) nm, the pixel array was 256 × 256, the dwell time was 10 ms/pixel, and the secondary ion intensities were summed over four planes for the image shown in Fig. 1(a). So, the total dose was \( 1.9 \times 10^{17} \text{ ions/cm}^2 \) or \(~3 \times 10^6 \text{ ions/pixel} \). The sputtering yield of 16 keV oxygen primary ions in the Si sample was calculated to be 0.8 at/ion by SRIM simulation. Hence, the total number of atoms sputtered per voxel is \(~2.4 \times 10^6 \) atoms. Taking the density of Si as \( 5 \times 10^{22} \text{ at./cm}^3 \), it can be shown that the estimated voxel size is about \( 39 \times 39 \times 30 \) nm\(^3\). The lowest possible dopant concentration that is theoretically possible is 1 dopant atom of the \(~2.4 \times 10^6 \) atoms or \(~2 \times 10^{16} \) at./cm\(^3\) or 0.4 ppm. The difference between this theoretical limit and the experimentally measured detection limit of \( 3 (\pm 2) \times 10^{17} \) at./cm\(^3\) is attributed to the limited useful yield resulting mainly from losses associated with ionization efficiency and the transmission of the spectrometer. In the present case, a detection limit of \(~3 \times 10^{17} \) at./cm\(^3\) would mean 15 B

Figure 3. (a) Schematic of the crystallographic relationships in the cross-section of pyramid as seen along the <011> direction and (b–d) 3D models of multilayer pyramid structure to illustrate the geometric effect of the slicing angle on the apparent enlargement of layer thickness. The colours indicate different layers as labeled in Fig. 2(a).
atoms per voxel to detect 1 B⁺ count. This corresponds to a useful yield of \(6.7 \times 10^{-2}\). For comparison, Migeon et al. [17] reported a useful yield of \(3.1 \times 10^{-3}\) for the detection of B⁺ in Si using O₂⁺ primary ions. The difference in useful yields is mainly because of the intrinsic differences in the transmission of the instruments and possible differences in analysis parameters such as mass resolution.

As an application example of this quantitative SIMS imaging method, carrier-selective passivating contacts on a textured c-Si wafer were analyzed by correlatively combining TEM and SIMS imaging. A schematic cross-section of the solar cell sample is shown in Fig. 2(a). As can be seen, the surface of textured c-Si substrate is passivated with a thin layer of SiOₓ (~1 nm) and an amorphous hydrogenated boron-doped SiCₓ layer (~30 nm). The presence of carbon in the SiCₓ layer enables (i) to tune the optical properties of the layer, (ii) improve the resilience of the layer to blistering due to hydrogen effusion occurring during subsequent annealing steps, and (iii) enhance its stability to wet chemistry processes used during the solar cell fabrication procedure [10]. The diffusion of dopants from the SiCₓ layer to the c-Si wafer during the annealing step contributes to a reduction of charge carrier recombination at the contact. The SiCₓ layer is strongly doped with boron in order to provide hole-selectivity, establish good contact to the external metallization, and to act as a dopant source for diffusion through the interfacial oxide and into underlying c-Si wafer.

A sample annealed at 850 °C was used for the present study. An SE image of the textured wafer surface of the sample is shown in Fig. 2(b). The (111) facets of the pyramids exposed after the texture etch are clearly visible. To evaluate the cross-sectional features, a STEM–HAADF image of a cross-section of a typical pyramid is shown in Fig. 2(c). The Pt layer was deposited during the FIB preparation to protect the surface from ion beam damage during the sample preparation. The SIMS images of the cross-section sample are shown in Figs. 2(d) and 2(e). Figure 2(e) was acquired at exactly the same conditions as Fig. 1(a) such that the boron concentration in the c-Si
substrate can be quantified from the secondary ion intensity using the calibration curve shown in the inset of Fig. 1(b).

Before evaluating the dopant concentrations, it is necessary to consider the crystallographic and geometric aspects of the pyramids in textured Si. The facets of the pyramids are (111) type which are formed on the [100] oriented Si wafer. A schematic illustration of the angular relationships between the facets and the substrate in a cross-section viewed along the ⟨011⟩ axis is shown in Fig. 3(a). The well-defined angular relationships allow assessing concentration variations of dopants along different crystal directions. However, the pyramid has to be precisely oriented at the time of sample preparation to subsequently determine the concentration variation along different crystallographic directions. This is a more elaborate investigation for future work and beyond the scope of the present study. Another aspect associated with the geometry of the pyramids is the influence of cutting angle on the apparent layer thicknesses as shown in Figs. 3(b)–3(d). The cutting plane results in dramatically different apparent layer thicknesses according to its angular relationship with individual facets locally. Note that the total thickness of the passivation layers in the analyzed sample is ~30 nm as shown schematically in Fig. 2(a). However, the pixel size in Fig. 2(e) is 39 nm, i.e., the entire multilayer stack is smaller than a single pixel in the image. However, we see the boron-containing layer to be extended over nearly 1 µm in Fig. 2(e). In addition to diffusion of B, there are two other reasons for this. One is the relatively large probe size (400 nm) and the other is the geometric projection effect due to the cutting angle as illustrated in Figs. 3(b)–3(d). However, this effect does not impact the present investigation as the facets oriented oblique to the surface (identified by white arrows in Fig. 2(d)) are excluded from the analysis.

To determine the dopant concentration profile in the c-Si substrate beneath the passivating contact layers, the line scans white arrows in Fig. 2(d)) are excluded from the analysis. For the investigated concentration range, a nonlinear relationship between $^{11}$B$^+$ intensity and the concentration was observed. A SIMS detection limit of 3 (±2) × $10^{17}$ at./cm$^2$ in the imaging mode with 39 nm pixel size was determined for the used experimental conditions. The method was demonstrated using an example of application with the passivating contact stack of an annealed textured solar cell material.

Conclusions

A method to rapidly and quantitatively image dopant distribution using SIMS is demonstrated. The method is based on the cross-section analysis of a reference sample with a known concentration profile. For the investigated concentration range, a nonlinear relationship between $^{11}$B$^+$ intensity and the concentration was observed. A SIMS detection limit of 3 (±2) × $10^{17}$ at./cm$^2$ in the imaging mode with 39 nm pixel size was determined for the used experimental conditions. The method was demonstrated using an example of application with the analysis of dopant concentration in c-Si adjacent to a passivating contact stack of an annealed textured solar cell material. Because of the pixel nature of the SIMS images, a locally higher concentration at sub-pixel length scale cannot be captured, which is an inherent limitation of the SIMS imaging technique. Hence, the concentrations determined provide average values pixel by pixel.

Supplementary material

The supplementary material for this article can be found at https://doi.org/10.1557/mrc.2019.89.

Acknowledgments

This work was funded by the Luxembourg National Research Fund (FNR) through grant INTER/SNF/16/11536628 and by the Swiss National Science Foundation (SNF) within the bilateral project NACHOS. Prof. Roger Webb (Ion Beam Centre, Surrey, UK) is thanked for discussions related to ion implantation parameters used for the reference sample. Brahime El Adib (LIST) is thanked for his technical assistance with SIMS depth profiling.
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