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Overcoming the quantification barrier in reverse tip sample scanning spreading resistance microscopy for efficient nanoscale carrier profiling

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Abstract

The quick tip exchange capability of reverse tip sample (RTS) scanning probe microscopy (SPM) provides significant efficiency benefits over conventional SPM, especially in high-force modes like scanning spreading resistance microscopy (SSRM). While prior developments in RTS SSRM have addressed conductive diamond probe chip fabrication, sample preparation for electrical SPM measurements, and qualitative measurement capabilities, the challenge of achieving quantitative carrier profiling remained unresolved, primarily due to focused ion beam (FIB) damage that is introduced during RTS sample preparation. In this work, we present two different FIB-based RTS sample preparation protocols for mounting multiple samples on a single tipless cantilever. The core postulation is that co-mounting the target and calibration samples together ensures that they undergo identical ion beam exposure, thereby enabling accurate SSRM quantification. The first protocol employs sequential mounting of individual samples, while the second involves bonding the samples together, polishing their surfaces, and then, mounting them as a single, unified assembly. It is also worth noting that the latter protocol supports a sample layout that enables quantitative analysis to be done on each individual scanline, which can reduce the impact of measurement artefacts resulting from tip degradation or just unstable tip-sample contact during scanning. Quantitative validation of RTS SSRM for both preparation protocols was accomplished by implementing two different staircase calibration samples, comprised of layers having well-defined carrier concentrations. Finally, mounting a calibration sample alongside an actual device sample confirmed that RTS SSRM delivers results equivalent to those obtained with conventional SSRM, affirming its quantification reliability for practical applications. By establishing quantitative SSRM in RTS configuration, this work addresses a critical gap and further positions RTS SPM as a valuable approach for advancing electrical SPM characterization.

1. Introduction

In nanoelectronics, scanning spreading resistance microscopy (SSRM) has been a pivotal characterization technique for nearly three decades now [1, 2], as it enabled quantitative nanometer-scale two-dimensional carrier profiling of nanodevice structures [3–7]. This quantitative capability has been made possible by a calibration procedure introduced by Vandervorst *et al* [8], which compensates for the contact, probe, and back-contact resistance contributions in the measurement output, that are difficult to determine accurately. In this approach, an additional reference, so-called ‘staircase’, calibration sample containing layers with well-defined carrier concentrations must first be measured with the same probe and under the same operating conditions as the target sample. The resulting calibration curve is then used to convert the spreading resistance maps of the target sample into quantitative carrier concentration maps. It is important to note, however, that having to switch back and forth between calibration

and target samples makes quantitative SSRM a rather low throughput technique. Additionally, for optimal measurement performance extremely high tip-sample contact pressures (in the order of 10 GPa) are needed to induce the silicon β -Sn phase transition [9, 10]. Such pressures inevitably cause gradual wear, even of diamond-based probes, leading to degraded spatial resolution and reduced quantification accuracy over time [11]. As a result, in quantitative SSRM, tips are replaced frequently. Each replacement is not only time-consuming by itself, as it is typically performed manually, but also requires repeating the full calibration procedure. The operator must re-engage both the calibration sample and the target sample, re-locate the regions of interest, and again ensure consistent measurement conditions, all of which further reduce the throughput.

Recently, the reverse tip sample (RTS) scanning probe microscopy (SPM) concept has been proposed as an alternative approach to conventional SPM, aimed at improving the efficiency of predominantly tip-invasive operational modes [12]. In the RTS configuration, the sample is mounted on a tipless cantilever, while the tips are integrated into a nanofabricated RTS probe chip that takes the position traditionally held by the sample and contains thousands of individual tips. This arrangement provides an effectively unlimited supply of tips and enables rapid tip replacement without interrupting measurements, thus holding significant promise to improve the operational efficiency of quantitative SSRM. Indeed, RTS probe chips with conductive diamond tips have already been developed for RTS SSRM applications [12, 13]. Moreover, qualitative measurements have also been done on a diverse range of samples, including multi-wall carbon nanotube interconnects, memory holes, GaAs nanoridge structures, FinFET devices, and most importantly, staircase calibration samples [2, 13–15]. Therefore, it is certainly possible to mount a staircase calibration sample on one cantilever and a target sample on another, which raises the question of why fully quantitative RTS SSRM measurements have not yet been demonstrated. The reason is that these previous demonstrations have relied on RTS sample preparation protocols which are based on focus ion beam (FIB) processing [14]. The issue here is that during FIB milling, Ga ions can disrupt the crystal lattice, creating an amorphous layer, and may even become implanted in the sample, altering its local electrical properties which will hinder a reliable quantification [16, 17]. A potential solution, recently proposed by Pandey *et al* [18], could be to subject both the calibration and target samples to identical FIB processing, which could then be followed up by the synchronized removal of FIB-damaged layers from both, with the Scalpel-SPM technique. However, if the samples are mounted on separate cantilevers for RTS measurements the depth-matched material removal on both would be challenging. And this is assuming that the two independently prepared samples would exhibit identical FIB-damage depth profiles, which is a condition that would be nearly impossible to guarantee. Moreover, switching between separate cantilevers for the calibration and target measurements would negate much of the throughput advantage offered by the RTS approach.

To address this challenge, in this paper, we introduce two novel FIB-based protocols for RTS sample preparation, which enable co-mounting of the target and calibration samples on a single tipless cantilever. This strategy ensures that both samples are subjected to identical ion beam treatments, eliminates the need for sample switching during quantification, and allows for consistent tip-sample interactions during measurements, as the force feedback is governed by the same cantilever spring constant and deflection sensitivity. The accuracy of carrier concentration quantification using both protocols was assessed, yielding results consistent with conventional SSRM. Furthermore, we show that one of these also allows for a sample arrangement that can support robust scanline-by-scanline SSRM quantification. Altogether, this work demonstrates, for the first time, fully quantitative SSRM using the RTS configuration, addressing the final obstacle to establishing RTS SSRM as a reliable, high-throughput alternative to conventional SSRM.

2. Single-scan-frame (SSF) and single scanline (SSL) quantification in RTS configuration enabled by sequential sample transfer (SST) and combined sample transfer (CST) sample preparation

As noted above, one major reason for low throughput in quantitative SSRM is that the standard workflow involves scanning the calibration sample, retracting the tip, and then repositioning it to engage with the target sample, as shown in figure 1(a). In addition, to ensure that the tip shape has remained unchanged during measurement, it is recommended to measure the calibration sample again afterward. Therefore, a more efficient approach is to eliminate the need for tip repositioning by placing the calibration and target samples close enough to each other such that both are within the atomic force microscope (AFM) XY piezo range. In this way both samples could be captured in a single SSRM scan, and it would allow for quantification to be done from that one scan, as illustrated in figure 1(b). This method

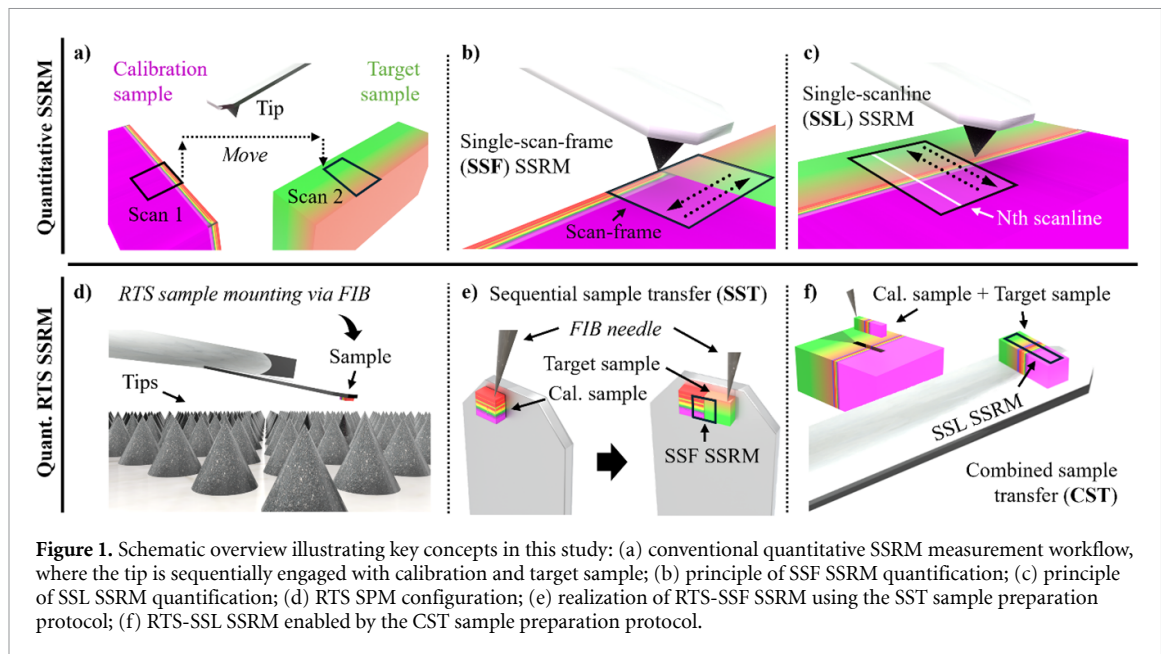


Figure 1. Schematic overview illustrating key concepts in this study: (a) conventional quantitative SSRM measurement workflow, where the tip is sequentially engaged with calibration and target sample; (b) principle of SSF SSRM quantification; (c) principle of SSL SSRM quantification; (d) RTS SPM configuration; (e) realization of RTS-SSF SSRM using the SST sample preparation protocol; (f) RTS-SSL SSRM enabled by the CST sample preparation protocol.

will be from here on out referred to as SSF quantification. Previous works by Coq Germanicus *et al* [19], and by Aldmaier *et al* [20], pursued this strategy not only for throughput gains, but also to reduce measurement uncertainties related to the probe condition and the scanning parameters. In that regard, for even better measurement reliability, samples can also be arranged so that during each scanline the tip traverses all the layers of the calibration sample before entering the region of interest (ROI) in the target sample, as shown in figure 1(c). This configuration makes it possible for quantification to be done on each individual scanline, a method we coin as SSL quantification. Under optimal SSRM measurement conditions, where the tip-sample electrical contact remains stable throughout the scan, SSL and SSF quantification would yield identical results. However, if there are electrical contact instabilities during the scan, the resulting measured resistance fluctuations will be reflected in both the calibration and the target sample data, which could compromise SSF quantification, whereas, in theory, SSL quantification accuracy will be preserved. It should be noted, however, that these concepts can be challenging to implement in practice, as one needs to keep the gap between the two samples small enough to avoid smearing artifacts and accelerated tip wear. In this work, we adopt RTS SPM configuration (figure 1(d)) for realizing both SSF and SSL SSRM. Typically, RTS sample preparation for electrical SPM modes, such as SSRM, utilizes a FIB—based process [14], involving protection and isolation of the ROI, sample pickup and transfer, attachment to a tipless cantilever using platinum (Pt) deposition, and subsequent side milling to flatten the surface that is to be measured. This final milling step has historically restricted the use of RTS for quantitative SSRM, due to ion beam-induced damage. To overcome this, two new RTS FIB-based sample preparation strategies have been developed, wherein the target and calibration samples are mounted side by side on the same cantilever and flattened simultaneously in a single ion beam milling session, ensuring equivalent ion beam exposure on both. These techniques are termed RTS SST and RTS CST and will be discussed in detail in the next section. SST was designed to facilitate SSF quantification, while CST enables SSL quantification, as depicted in figures 1(e) and (f), respectively.

3. RTS multi-sample preparation protocols for quantitative SSRM

SST and CST RTS sample preparation protocols were developed and executed using either the Helios 450 or Helios 460 dual-beam FIB/SEM (scanning electron microscope) systems from Thermofisher, equipped with a tungsten probe needle micromanipulator and a Pt deposition module. The tipless cantilevers used for mounting the samples were in-house fabricated, electroplated nickel cantilevers with a nominal spring constant of 27 N m^{-1} and dimensions of $225 \text{ }\mu\text{m}$ (length), $50 \text{ }\mu\text{m}$ (width), and $5 \text{ }\mu\text{m}$ (thickness). While these custom cantilevers were employed in the present work, any commercially available conductive tipless cantilever would be equally suitable. All RTS SSRM measurements were conducted under ambient conditions with a boron doped diamond coated RTS probe chip [12], using a Bruker Dimension Icon PT AFM equipped with an SSRM application module.

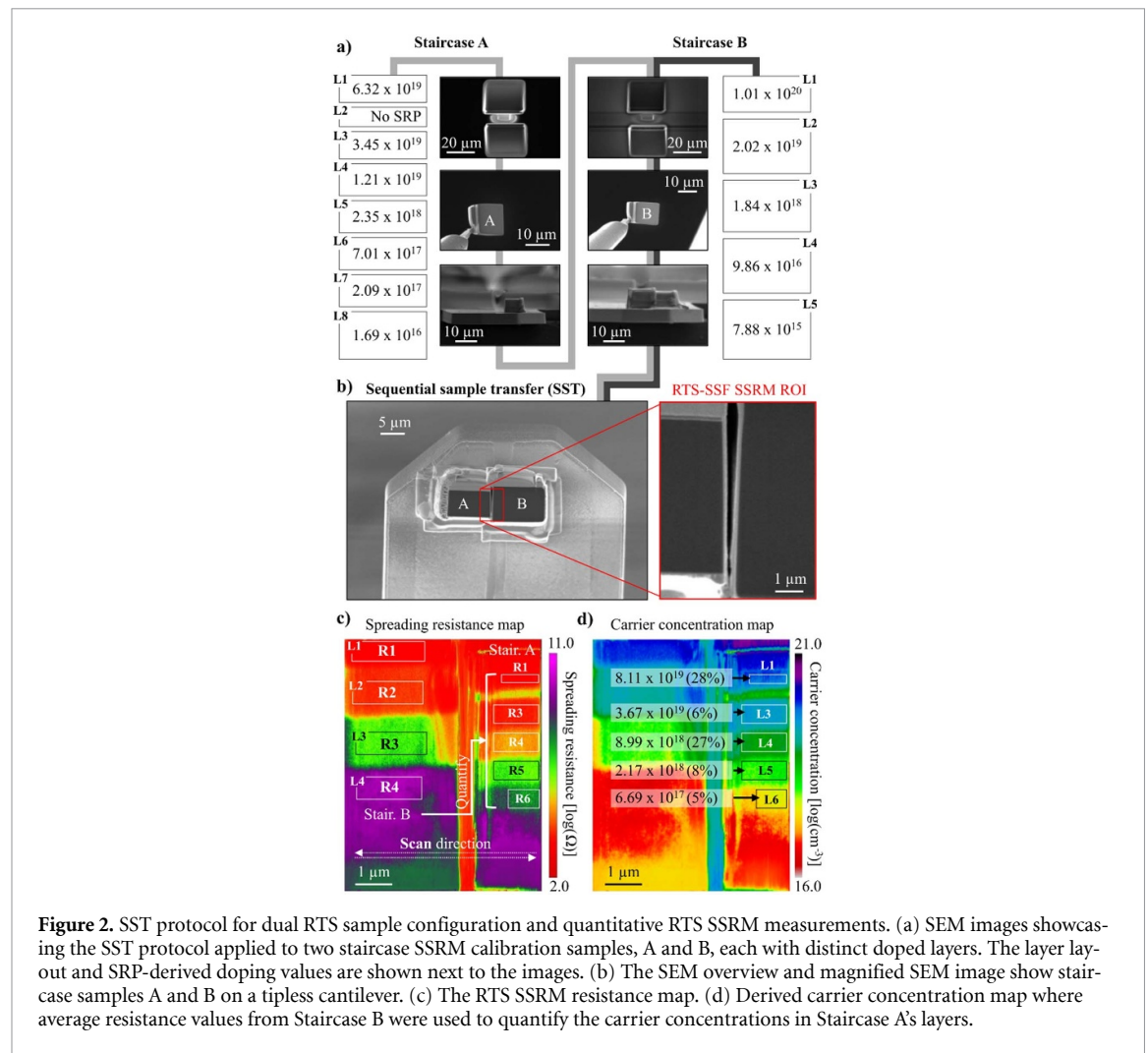


Figure 2. SST protocol for dual RTS sample configuration and quantitative RTS SSRM measurements. (a) SEM images showcasing the SST protocol applied to two staircase SSRM calibration samples, A and B, each with distinct doped layers. The layer layout and SRP-derived doping values are shown next to the images. (b) The SEM overview and magnified SEM image show staircase samples A and B on a tipless cantilever. (c) The RTS SSRM resistance map. (d) Derived carrier concentration map where average resistance values from Staircase B were used to quantify the carrier concentrations in Staircase A's layers.

3.1. SST procedure and quantitative RTS-SSF SSRM measurements

In essence, the SST protocol relies on a simple premise that if a process can be done once, it can be done multiple times. Specifically, the FIB-based protocol for preparing cross-sectional RTS samples is performed twice on the same cantilever, with minor additional steps [14]. A series of SEM images in figure S1 showcases the SST process which begins with depositing a protective Pt layer over the ROI, followed by milling two large trenches on either side and performing an undercut beneath the ROI. The sample is then lifted from the die using a probe needle micromanipulator and transferred to a tipless cantilever, where it is secured with Pt deposition. The side that will be facing the second sample is straightened to ensure a snug fit between the two. The second sample is then prepared and transferred in the same way as the first, positioned directly adjacent to it. Once both samples are attached, material is milled from the front side until the target position is reached. To evaluate if the SST protocol could enable quantitative SSRM analysis in RTS configuration, we used two different p-type doped Si staircase samples, hereafter referred to as Staircase A and Staircase B. The doping concentrations for all layers, with the exception of layer 2 (L2) of Staircase A, in both samples were measured using spreading resistance profiling (SRP). These reference values are presented in figure 2(a), alongside SEM images representative of three main steps (ROI isolation, sample pickup, and sample transfer) of the discussed SST procedure for each sample. The idea was to use one calibration sample to quantify another and compare the results with SRP values. Staircase A was chosen as the target sample to prevent extrapolation, given its narrower doping range. SEM image of the final SST result is shown in figure 2(b), with the interface between the two samples further highlighted in the zoomed-in inset. These images confirm that the gap between samples was small enough to enable simultaneous capture of both samples within a single SSRM scan frame (SSF method). The measurement procedure began with repeated SSRM scans at a higher load force to synchronously remove FIB-damaged material (Scalpel-SPM) from both the calibration and target sample, in accordance with the above discussed hypothesis. This was done until

clear resistance contrast could be seen across the first four layers of Staircase B, which was the indicator that the FIB-affected material has been sufficiently removed. Quantitative RTS-SSF analysis was then performed on the subsequent SSRM scan, shown in figure 2(c). The calibration curve was constructed using measured resistance values (R1, R2, R3, R4) and known carrier concentrations from Staircase B. The resulting carrier concentration map is presented in figure 2(d). Measured doping values for layers 1 through 6 (excluding L2) of Staircase A were close to the SRP reference values, with calculated linear percentage errors of 28%, 6%, 26%, 8% and 5%, respectively. Combined, this yields an average error of 15%, which is well within typical accuracy limit of conventional SSRM, around 25% [8]. Accordingly, these results confirm that the SST protocol enables quantitative RTS-SSF SSRM measurements.

3.2. CST procedure and quantitative RTS-SSL SSRM measurements

While the SST protocol described above supports RTS-SSF SSRM quantification, the CST protocol was developed as an epoxy-based alternative that not only offers the same capability but also RTS-SSL. The sample arrangement for this in the case of calibration staircase A–B samples is schematically illustrated in figure 3(a), again with their respective SRP-measured layer carrier concentration values.

Here, the CST protocol begins with thorough cleaning of both Staircase A and B samples with acetone and isopropanol to remove any surface contaminants. Next, the two samples are carefully glued together face-to-face, with particular attention to minimizing the gap between them, following a procedure similar to that described by Coq Germanicus *et al* [19]. As depicted in figure 3(b), the bonded assembly is then polished to ensure both a minimal height difference due to any slight misalignment and a low surface roughness of the ROI. Optical microscopy images acquired after the polishing step are shown below the schematic. Subsequently, the samples are transferred into the FIB/SEM system, where FIB processing is performed according to the established top-view RTS sample preparation protocol [14]. Figure 3(c) shows SEM images corresponding to several major FIB-steps in this protocol, including sample protection and isolation, pickup and transfer, and the final product. It should be noted that the CST takes longer than SST (four hours versus three) due to the additional pre-FIB steps. However, if multiple RTS samples need to be prepared from a single sample combination, the CST protocol will be more efficient as the bonded and polished sample assembly can be re-used.

After the CST Staircase A–B sample was prepared, RTS SSRM measurements were conducted similarly to those performed on the SST-prepared sample. First, the FIB-damaged layers were removed, and then another SSRM image was captured to be used for quantification. However, this time around, the scan direction was set to be perpendicular to the layers of both calibration samples, so that all resistance values would be captured within each SSRM scanline. Figure 4(a) shows the resulting spreading resistance map. As with the RTS-SSF analysis performed on an SST prepared sample, the aim here was to quantify the layers of Staircase A using those of Staircase B. However, since the CST approach facilitates both SSF and SSL analyses, this SSRM image served as an ideal platform for comparing the two. For RTS-SSF quantification, resistance values R1 to R4 from Staircase B were averaged from the regions illustrated with the white dashed bounding boxes in figure 4(a) to construct the calibration curve and subsequently convert the resistance map into a carrier concentration map. Conversely, the RTS-SSL quantification involved constructing a separate calibration curve for each of the 216 scanlines present in the image, with one highlighted in figure 4(a). For a given scanline, this was done by collecting and averaging resistance values from all pixels associated with each layer of Staircase B but limited to the same bounding boxes utilized in the RTS-SSF analysis to ensure a fair comparison. The obtained calibration curve was then used to convert all pixels in that scanline from resistance values to carrier concentration values. The procedure was repeated across all scanlines to generate a complete carrier concentration map, which is presented in figure 4(b) alongside the map obtained via RTS-SSF quantification. Average doping values for layers of Staircase A were then extracted from both carrier concentration maps and compared with their corresponding reference SRP values to calculate the quantification errors, which are summarized in the table in figure 4(c). Both quantification methods produced errors that were comparable within the margin of rounding: 32%, 21%, 8%, 22%, and 25%, for L1–L6 (except L2). With the average error of 22%, these results are again within the accuracy range of conventional SSRM, further validating the feasibility of performing quantitative SSRM in the RTS configuration, but now also on CST prepared samples. Furthermore, just based on visual inspection, RTS-SSL maps have noticeably more uniform doping contrast within layers. This trend is particularly evident in areas where electrical contact was less stable, such as the left-edge region of the SSRM image which contains an edge scan artifact. In standard SSRM practice, such regions are typically excluded from quantification. This approach was also adopted for the above-described comparative analysis, again as indicated by the dashed bounding boxes in figure 4(a). Likewise, average doping values for Staircase A layers used to compute the errors for both analysis methods shown in the table were also taken by avoiding this region (solid-line bounding boxes

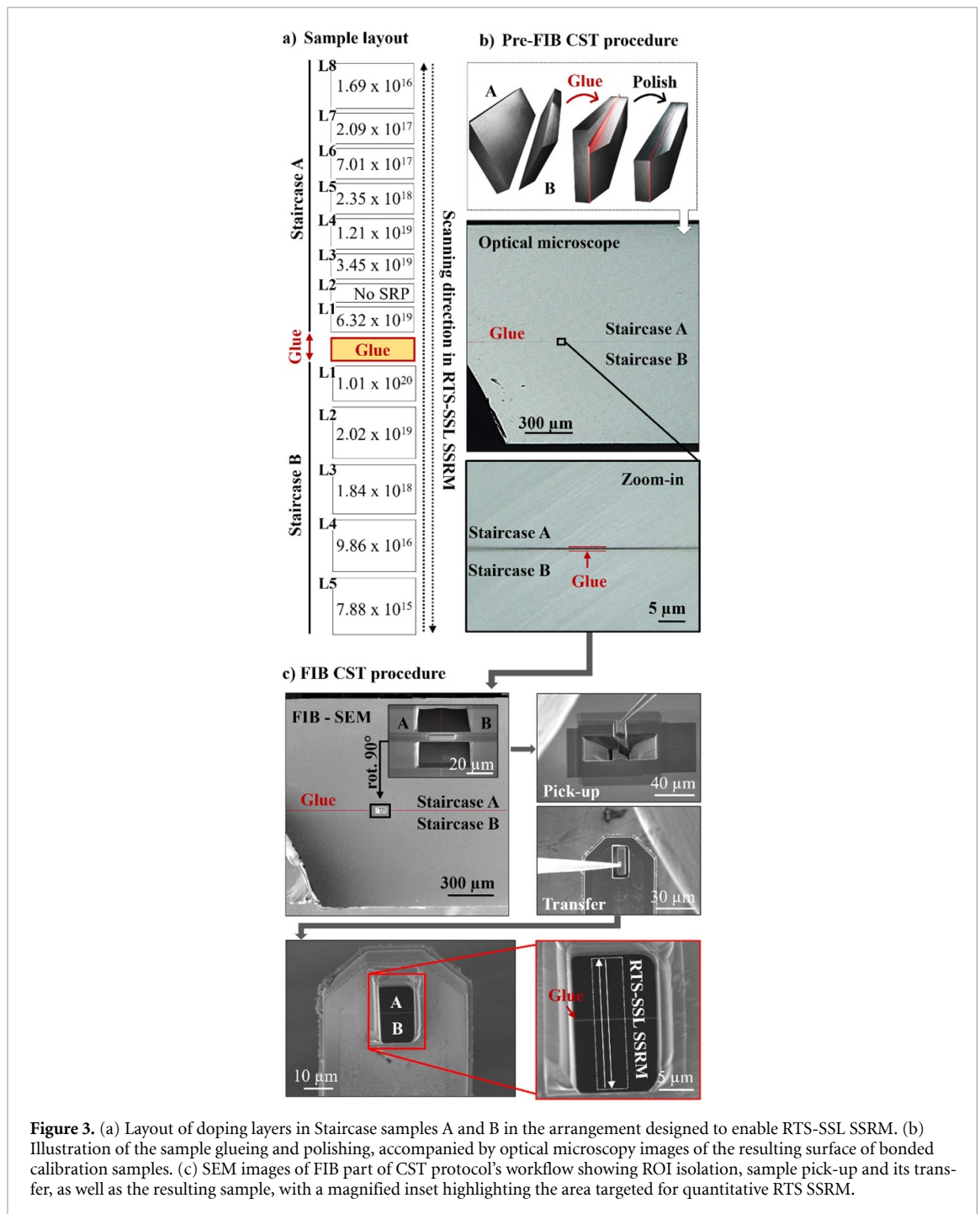


Figure 3. (a) Layout of doping layers in Staircase samples A and B in the arrangement designed to enable RTS-SSL SSRM. (b) Illustration of the sample glueing and polishing, accompanied by optical microscopy images of the resulting surface of bonded calibration samples. (c) SEM images of FIB part of CST protocol's workflow showing ROI isolation, sample pick-up and its transfer, as well as the resulting sample, with a magnified inset highlighting the area targeted for quantitative RTS SSRM.

in figure 4(b)). Had this not been the case, the error comparison between RTS-SSF and RTS-SSL would have revealed more significant differences. Figure 4(d) shows close-ups of the resistance map in the artefact region for layers of Staircase A, along with calculated error maps. Above each error map both the means of carrier concentration values and the means of pixel-wise errors for that layer are indicated. These error maps were constructed by comparing the carrier concentration value of each pixel to the value obtained from SRP measurements for that given layer. This was done because calculating an error using mean carrier concentration value of the layer can mask some of the local variations within it. For example, and in the case of RTS-SSF layer 6, the mean carrier concentration value is $7.33 \times 10^{17} \text{ cm}^{-3}$, and the calculated error on this mean is approximately 5%, while the pixel-wise mean error for the same layer from the error map is 47%. Across all the layers, the pixel-wise mean errors from RTS-SSF maps are, on average, 17% greater than the ones obtained from RTS-SSL maps. Moreover, the larger error values are clustered where resistance increases rapidly due to poor tip-sample contact. In contrast, the RTS-SSL error maps show significantly less indication of this measured resistance variation. Therefore,

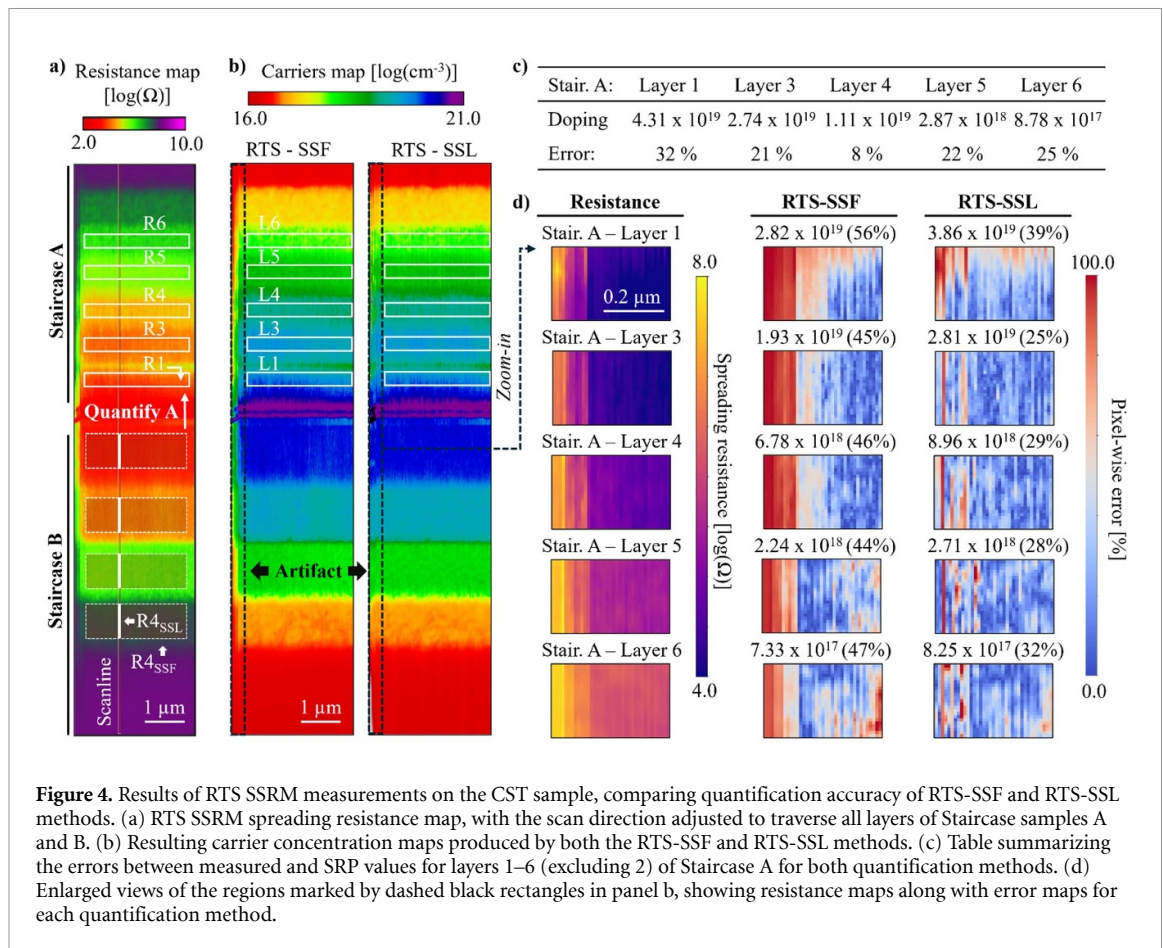


Figure 4. Results of RTS SSRM measurements on the CST sample, comparing quantification accuracy of RTS-SSF and RTS-SSL methods. (a) RTS SSRM spreading resistance map, with the scan direction adjusted to traverse all layers of Staircase samples A and B. (b) Resulting carrier concentration maps produced by both the RTS-SSF and RTS-SSL methods. (c) Table summarizing the errors between measured and SRP values for layers 1–6 (excluding 2) of Staircase A for both quantification methods. (d) Enlarged views of the regions marked by dashed black rectangles in panel b, showing resistance maps along with error maps for each quantification method.

the RTS-SSL method does offer better preservation of quantification accuracy compared to RTS-SSF in challenging SSRM images which are, in fact, common in practice.

3.3. Comparative analysis of quantitative RTS SSRM and conventional SSRM on solar cell (SC) device structure

Finally, to evaluate the practical applicability of quantitative RTS SSRM under real-world conditions and directly compare its performance to conventional SSRM, one of the staircase calibration samples was replaced with an actual, unknown target sample. Calibration sample Staircase A was combined with a SC device structure, consisting of a blanket Si substrate overlaid with a heavily doped polycrystalline Si (poly-Si) layer. Dopant diffusion from the poly-Si into the underlying crystalline Si substrate was observed, and the goal was to quantitatively assess the dopant concentration in the uppermost region of the crystalline Si substrate. Two RTS samples were prepared using both SST and CST sample preparation protocols, with SEM images of both shown in figure 5(a). Figures 5(b) and (c) present the results from the RTS SSRM measurements performed on these samples. Additionally, SSRM was conducted in a conventional configuration on the same sample combination, with results displayed in figure 5(d). Figure 5(e) shows three doping profiles extracted from converted carrier concentration maps along the paths indicated by black arrows in panels (b) and (c), corresponding to standard SSRM and RTS SSRM done on two above-described RTS samples. Excluding the poly-Si layer, where quantification is not valid due to its matrix differing from that of the crystalline Si calibration staircase sample, minor variations in carrier concentration across the three SSRM measurements were observed. Importantly, all these variations again fall well within the established accuracy of SSRM in addition to carrier profiles sharing the same overall shape. In the standard SSRM measurement, a maximum carrier concentration of $8 \times 10^{18} \text{ cm}^{-3}$ was measured just beneath the poly-Si layer. The quantitative RTS SSRM measurements on SST- and CST-prepared samples yielded a concentration of $4 \times 10^{18} \text{ cm}^{-3}$ and $7 \times 10^{18} \text{ cm}^{-3}$, respectively. This result conclusively confirms that RTS SSRM is on par with standard SSRM for quantitative carrier profiling in device structures.

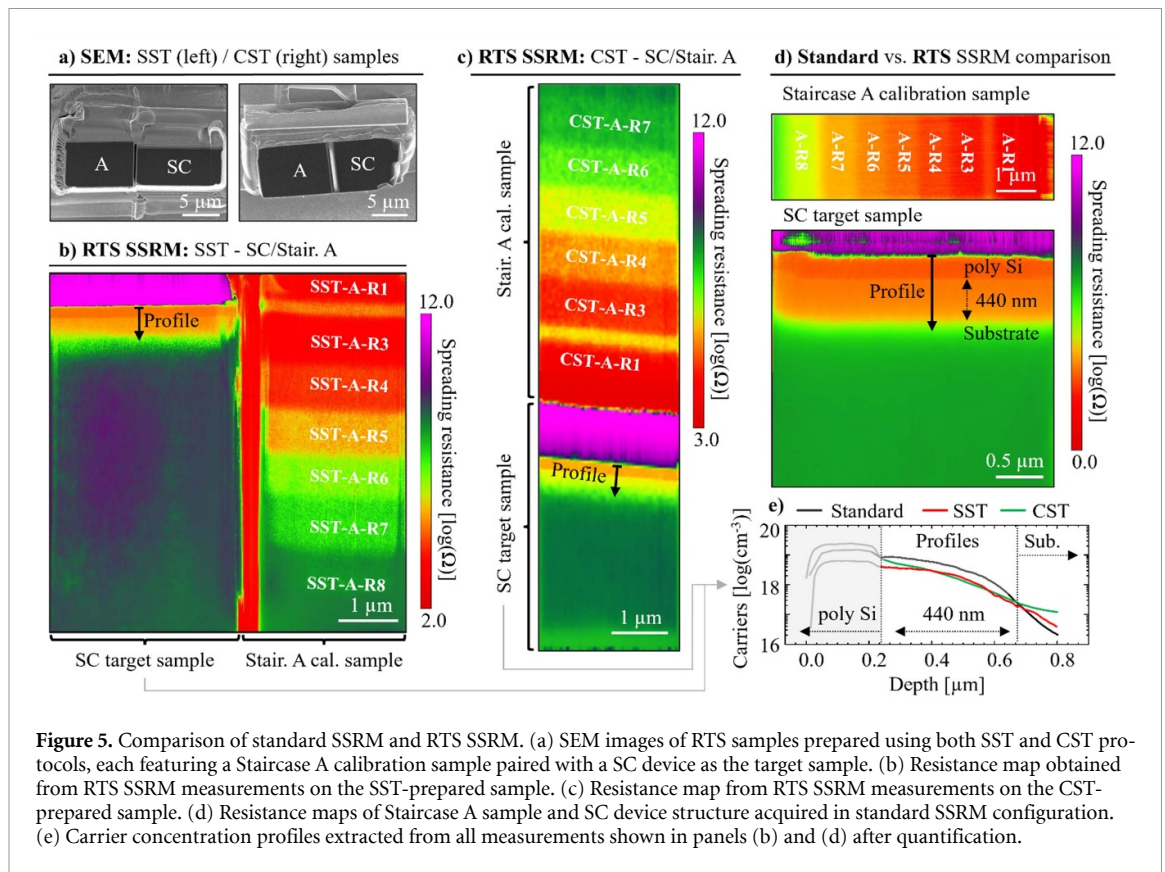


Figure 5. Comparison of standard SSRM and RTS SSRM. (a) SEM images of RTS samples prepared using both SST and CST protocols, each featuring a Staircase A calibration sample paired with a SC device as the target sample. (b) Resistance map obtained from RTS SSRM measurements on the SST-prepared sample. (c) Resistance map from RTS SSRM measurements on the CST-prepared sample. (d) Resistance maps of Staircase A sample and SC device structure acquired in standard SSRM configuration. (e) Carrier concentration profiles extracted from all measurements shown in panels (b) and (d) after quantification.

4. Conclusions

In summary, we have developed and validated two FIB-based RTS sample preparation protocols, SST and CST, to enable quantitative SSRM to be done in RTS configuration. Both were specifically designed to facilitate mounting of the target and calibration samples on a single tipless cantilever. The SST protocol involves placing samples one after the other, while the CST protocol employs first bonding the samples face-to-face, followed by surface polishing and subsequent extraction and mounting the samples as a unified assembly. In both protocols, the final FIB milling step is performed on both samples at the same time, ensuring they receive identical ion beam exposure and, as a result, matched FIB-induced damage depth profiles that can be removed using the Scalpel SPM technique. Moreover, having two samples together on one cantilever enables necessary quantification data to be collected in a single SSRM scan frame, or even within one scanline for CST-prepared samples. Consequently, in RTS SSRM there is no need for sample switching, unlike in conventional quantitative SSRM, which results in improved throughput and reliability. In our validation experiments, we used two different SSRM staircase calibration samples to measure carrier concentrations of layers in one sample using the calibration curve constructed from the other and then compared the results with SRP-obtained values for the same layers. Derived carrier concentrations from quantitative RTS SSRM on both SST- and CST-prepared samples remained within the established error margins of conventional SSRM. Furthermore, scanline-by-scanline quantification has proven effective in analyzing challenging data sets that might otherwise be excluded if only the conventional SSRM quantification workflow was employed. Lastly, applying quantitative RTS SSRM to a relevant SC device structure demonstrated that its performance matches that of conventional quantitative SSRM, but now with the added benefit of having rapid access to thousands of tips.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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