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Enabling focused ion beam sample preparation for application in reverse tip sample scanning probe microscopy

P. Lagrain *, K. Paulussen, E. Grieten, G. Van den Bosch, S. Rachidi, D. Yudistira, L. Wouters, T. Hantschel

Imec, Kapeldreef 75, B-3001 Leuven, Belgium

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ABSTRACT

Focused ion beam (FIB) has become a powerful tool for transmission electron microscopy sample preparation in the nanoelectronics industry and has in recent years also shown its benefits for specific preparation steps in electrical scanning probe microscopy (SPM). Most recently, a novel SPM approach – so-called reverse tip sample (RTS) SPM – has been proposed in which the position of sample and tip are switched compared to standard SPM; in RTS SPM the sample is attached to the end of a cantilever beam. To achieve this configuration, the region of interest must first be extracted from a substrate and then needs to be reliably fixed to the cantilever by FIB. Therefore, we have explored and developed dedicated FIB preparation methods for RTS SPM in this work. Our established procedures ensure a strong mechanical and good electrical connection of the sample to the cantilever for both cross-section and top view sample preparation. Furthermore, we introduce an approach for mounting samples from a full wafer size workflow. This paper presents the developed FIB procedures and discusses the quality and stability of all mounted samples and their electrical evaluation in RTS SPM.

1. Introduction

Focused ion beam (FIB) - integrated into state-of-the-art dual-beam FIB/scanning electron microscopy (SEM) systems - has become a powerful workhorse in the nanoelectronics industry (e.g. for sample preparation in transmission electron microscopy (TEM) [1–7] and for cross-section device inspection [8]) over the course of the past 25 years. More recently, it has also demonstrated its benefits as a specific preparation step for scanning probe microscopy (SPM) analysis; in particular in electrical SPM (ESPM) where it is today routinely used for establishing electrical back contacts by Pt deposition, marking the region of interest by placing etching markers, locally milling away material, and for fine polishing sample surfaces [9,10]. SPM uses a sharp tip mounted at the end of a cantilever beam to scan a sample surface while measuring various physical properties. As the tip gradually wears off during SPM measurements (in particular in ESPM where typically much higher forces are being used), frequent manual probe changes are required which are time consuming, cumbersome to do, and interrupt the ongoing experiment. To overcome this fundamental obstacle of classical SPM, the concept of reverse tip sample (RTS) SPM has recently been proposed [11] in which the normal position of tip and sample is switched - the tip is placed on the stage where normally the sample is located, and the sample is placed onto the cantilever. The main advantage of RTS SPM is that hundreds to thousands of tips can be integrated onto a tip array chip and that the tip changes can be done seamlessly within seconds without stopping the ongoing experiment. A crucial requirement for the successful application of RTS SPM is the availability of suitable methods for mounting the sample to be measured onto the cantilever beam. As FIB is already well established for TEM and ESPM sample preparation and is widely used in the nanoelectronics industry, it looks very promising for routine RTS SPM sample preparation. Therefore, we explored in this work the development of reliable and robust FIB procedures for RTS SPM and validated them on different test structures and evaluated the prepared samples in actual RTS SPM measurements. This paper presents and discusses optimized procedures for both cross-section and top view analysis. Furthermore, we demonstrate besides small sample sizes also first results on full wafer size sample preparation.

Fig. 1 illustrates the general principle for sample preparation used in this work whereby we leverage the well-known standard TEM preparation as a starting point and modified and extended it further to meet the specific requirements of RTS SPM analysis. The sample must be first

* Corresponding author.
E-mail address: pieter.lagrain@imec.be (P. Lagrain).

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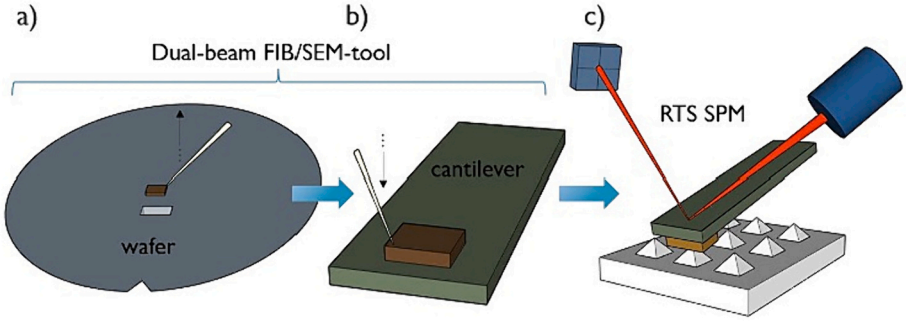

Fig. 1. Basic concept of FIB based sample mounting for RTS SPM: a) extract region of interest (ROI) from substrate, b) mount ROI sample chunk to cantilever end, c) load and measure cantilever mounted sample in RTS SPM mode.

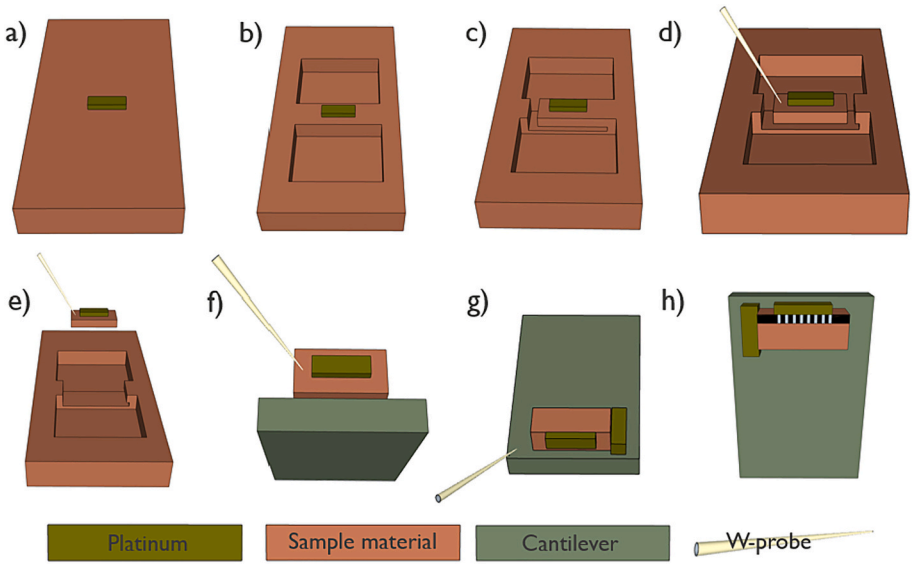

Fig. 2. Basic workflow of FIB based sample preparation for RTS SPM: a) deposit protective Pt, b) mill trenches, c) undercut, d) attach W-probe and cut chunk loose, e) lift-out chunk, f) position chunk at cantilever, g) attach chunk to cantilever and remove W-probe, and h) thin down chunk until ROI is reached.

extracted from a (wafer) substrate by FIB (Fig. 1a), then it is transferred and both mechanically and electrically well connected to a cantilever beam by means of FIB and a manipulator needle (Fig. 1b), and finally the cantilever-sample assembly is loaded into the SPM system for analysis in RTS SPM mode (Fig. 1c). This paper presents the two developed basic approaches and discusses them below: a combined FIB and gluing approach, and a more integrated and reliable FIB-only approach.

2. First approach – Combined FIB/epoxy

Earlier work on the preparation of TEM samples by FIB [3] already laid a foundation for this work and is used as a starting point for the RTS SPM sample preparation. Fig. 2 shows a schematic of the general workflow which we followed to mount samples on cantilever beam structures.

All steps were performed by a Helios 450 or Helios 460 dual-beam FIB/SEM system from Thermofisher equipped with a W-probe needle micromanipulator and a Pt deposition module. The cantilevers used in this work were inhouse fabricated Ni cantilevers with a nominal spring constant of 27 N/m (225 µm long, 50 µm wide, 5 µm thick). The steps shown in Fig. 2a through Fig. 2e are similar as for standard TEM sample preparation, with Fig. 2a showing the 1 µm thick protective Pt deposition as FIB hardmask at the ROI, Fig. 2b showing the milling of ~20 µm by 15 µm wide trenches with a depth of ~12 µm, Fig. 2c showing the performance of a trench wide undercut below the ROI, Fig. 2d indicating the attachment of a W-probe needle for the transfer action and the execution of a FIB cut to release the chunk from the substrate, and Fig. 2e showing the lift-out of the chunk. Note that the steps shown in Fig. 2f to Fig. 2h are specific to our RTS SPM case, with Fig. 2f depicting the positioning with respect to the cantilever, Fig. 2g showing the attachment of the chunk to the cantilever and the removal of the W-probe, and Fig. 2h highlighting the final chunk thinning until the ROI is reached. Note that the specific chunk dimension depends on the application case but is typically within 6–15 µm in length, 3–5 µm in width, and 4–8 µm in thickness.

Just as for TEM, one can also prepare two general types of samples for RTS SPM: cross-section samples where the surface to be measured is perpendicular to the die surface, and top view (often also referred to as plan view) samples, where the surface to be measured is parallel to the die surface.

For the cross-sectional procedure, a small piece of material is taken out of a functional die as shown in steps Fig. 2a through Fig. 2e. In this first approach, this chunk is attached to the sidewall of a Cu TEM grid and the W-needle is cut loose, similar as for the start of an actual TEM sample preparation. Note that we define the surface that we will eventually measure by SPM as the frontside, and the other side as the backside of the sample. By tilting the stage of the FIB-tool to 52°, it is now possible to flatten the back surface of the sample, which will act as the landing area on the tipless cantilever in this approach. In case the electrical backcontacting of the sample needs more attention, this

Table 1
Optimized FIB tool settings used for the steps described in Fig. 2.

Preparation step	Tool stage tilt (°)	Ion beam current (nA)
Protective Pt	52	0.23
Large trenches	52	21–49
Undercut	0	2.5
Cut loose	0	0.79
Lift-out	0	/
Positioning	0	/
Attachment	0	0.23
Thinning (30 kV)	52	0.08–0.79
Last thinning (5 kV)	55	0.12

configuration can also be used to deposit some Pt on the backside of the sample prior to mounting it on the cantilever. After reinsertion of the W-needle and attachment of the needle to the chunk, the sample is cut loose again from the Cu grid. The sample is then transferred to the tipless cantilever (Fig. 2f), which is vertically fixed to the sidewall of the FIB sample holder and is attached to the cantilever using Pt deposition. The sample is finished by milling away material from the front surface until the targeted measurement position is reached. All FIB steps are performed with an ion beam acceleration voltage of 30 kV. Note that some partial amorphization of the surface layers will always be present due to the interaction of the Ga FIB-beam with the crystalline sample material. Therefore, in the last step, the sample surface is trimmed down at 5 kV to reduce this beam surface damage [10,12].

Based on our experiments, Table 1 summarizes an optimized set of general tool parameters for the used FIB-system. Please note that these can vary on different tools or when different chunk dimensions are being used.

The top view sample preparation follows first the steps as shown in

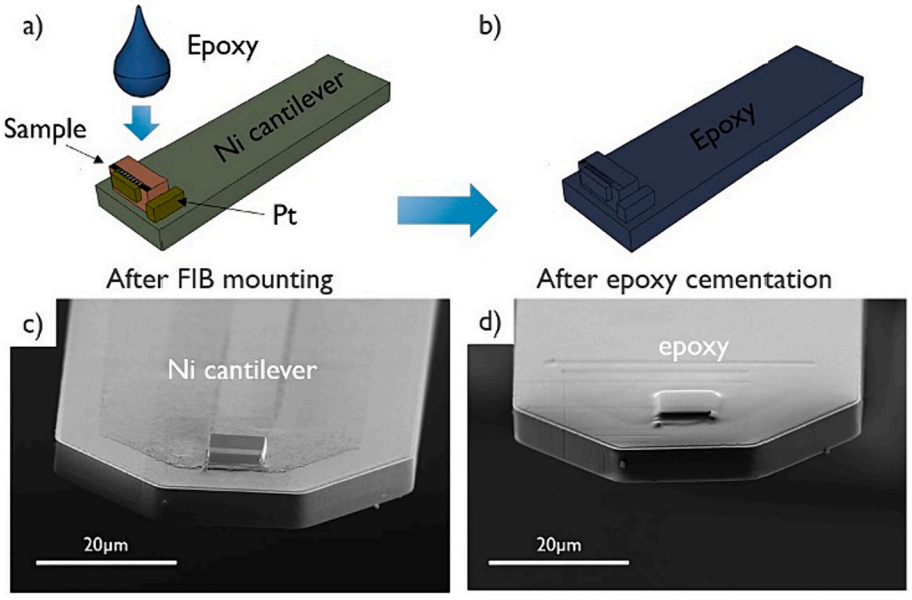

Fig. 3. Initial procedure for sample preparation needs epoxy cementation: schematic view of prepared sample a) before and b) after epoxy; SEM image of prepared sample c) before and d) after epoxy.

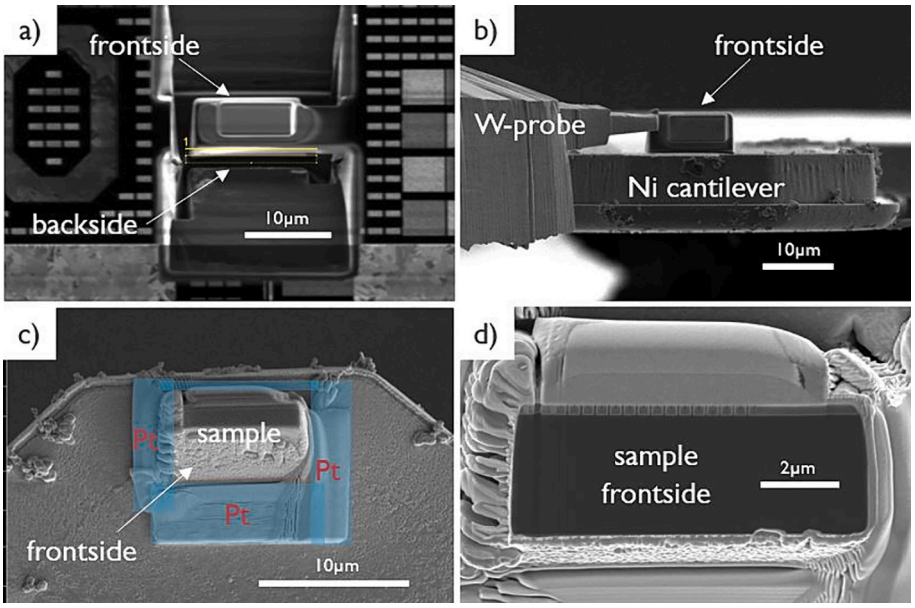

Fig. 4. Cross-section sample preparation: a) flattening back surface, b) attachment to cantilever, c) all-around Pt encapsulation, and d) end result.

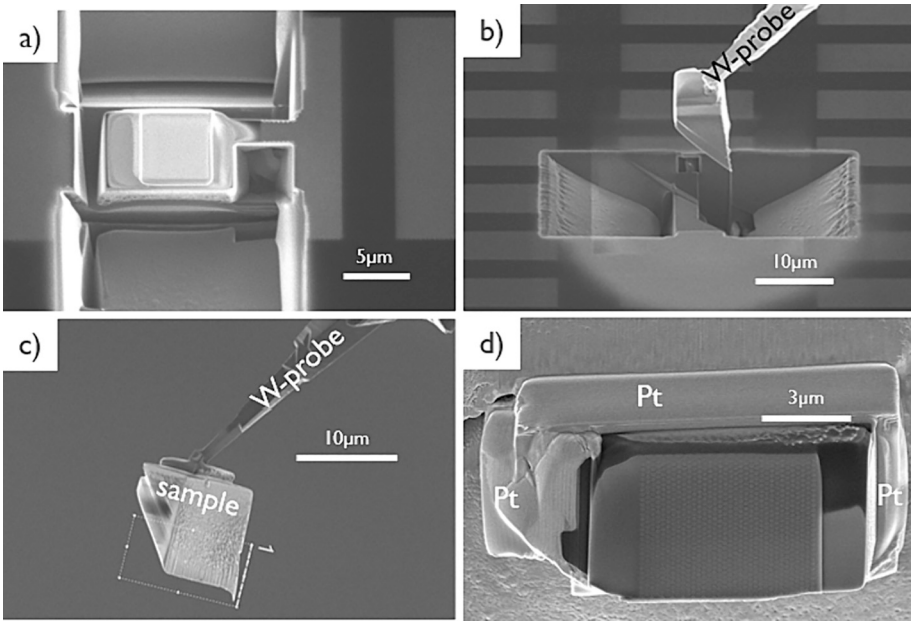

Fig. 5. Top view sample preparation: a) preparation of chunk, b) lift-out 90° rotated, c) configuration for flattening bottom surface, and d) end result.

Fig. 2a through Fig. 2c. Next, the sample is rotated by 90° to perform the lift-out step (Fig. 2d-2e). Note that now an extra step is added to flatten the bottom surface of the chunk, which is serving as the landing area. Rotating the W-needle 180° around its own axis enables the removal of material from the bottom surface to flatten this landing area. More details of this procedure can be found below in section 3. Another difference with the cross-section procedure is that the tipless cantilever is attached horizontally to the surface of the tool stage and that the sample is transferred directly from the die to the cantilever (Fig. 2g-2h) without the use of a Cu TEM grid. The total sample preparation time is about 120 min.

Initial experiments however showed that this local FIB based mechanical fixation is not sufficient to perform contact-mode SPM measurements afterwards, especially for the cross-section samples. To solve this problem, an extra preparation step is added in which the samples are manually cemented in an epoxy (Loctite Super Glue3) to guarantee the reliable mechanical strength of the samples.

Fig. 3 shows the general principle of this extra step whereby a small amount of epoxy fixed to the end of a sharp needle is applied to the cantilever end (Fig. 3a and c) and spreads around the sample area (Fig. 3b and d) which results in a stable mechanical fixation of the sample to the cantilever. While this approach works, it also causes some drawbacks. Firstly, the manual application of the epoxy on the sample under an optical microscope bears the significant risk of losing the sample altogether while performing it. Secondly, during this cementation the sample surface is covered by a thin epoxy layer itself. Note that on one hand we observed that this epoxy surface layer can be easily removed by performing a few low-force contact SPM scans. On the other hand this still generates extra scan debris around the measurement area which can affect the measurement result.

3. Second approach - FIB-only

Since the epoxy cementation step has substantial drawbacks, a second - FIB-only - procedure was developed. This second approach leaves out the intermediate fixation to a Cu TEM grid from the procedure, making it also more time-efficient.

For the preparation of cross-section samples, a tipless cantilever is again attached straight-up to the sidewall of the FIB sample holder. Steps Fig. 2a through Fig. 2c are still performed the same way. After performing the undercut (Fig. 2c) however, the stage is tilted to 52° again, so an extra step for flattening the back surface of the sample can be added. Note that, just as in section 2, we define the surface that we will eventually measure by SPM as the frontside, and the other side as the backside of the sample. This is shown more clearly in Fig. 4. By performing this step before lifting the chunk out of the die, there is no need anymore to attach the chunk to a Cu TEM grid. Fig. 4a shows this extra step.

When our back surface is flat, the chunk is lifted out of the die at 0° tilt and is immediately transferred to the cantilever at the same tilt angle, as seen in Fig. 4b. By depositing Pt on the right interface, the mechanical fixation is now sufficient to keep the sample in place, but this is not yet sufficient for high-force SPM measurements. Therefore, the cantilever is manually positioned flat on the sample holder instead of straight up. In this position, we are able to deposit blocks of about 1 µm thick Pt on all four sides of the sample to fully encapsulate it. Note that in this way, the mechanical fixation of the samples is strongly increased such that we are able to perform even the most strenuous SPM measurements later on without the use of an epoxy encapsulation step. This step also ensures that the surrounding Pt provides an electrical back-contact for the devices that are being probed. Fig. 4c shows the end result. To finish the sample, the cantilever is positioned straight up again, the stage is tilted to 52° and material is milled away until the desired end position is reached. Apart from locating our end position, this milling step also ensures that our sample surface has minimal topography and previously introduced debris is removed from the region of interest. Fig. 4d shows the end result. All actions are again performed with an ion beam acceleration voltage of 30 kV. As a very last step, we can again trim the sample surface down at 5 kV to reduce beam damage of the surface. Note that because the intermediate attachment to a Cu TEM grid is omitted in this procedure, samples can be made in about 90 min of preparation time compared to 120 min in the first approach. Furthermore, no extra treatments of the sample are necessary after this preparation.

For the top view samples, most of the preparation has stayed similar to the one in the initial procedure. The steps shown in Fig. 4a through Fig. 4c still apply. Fig. 5a shows a first difference, namely that some extra material has to be removed first, after which the lift-out happens 90° rotated compared to the cross-sectional case. Fig. 5b shows this rotated set-up. Another important difference is that the flattening of the

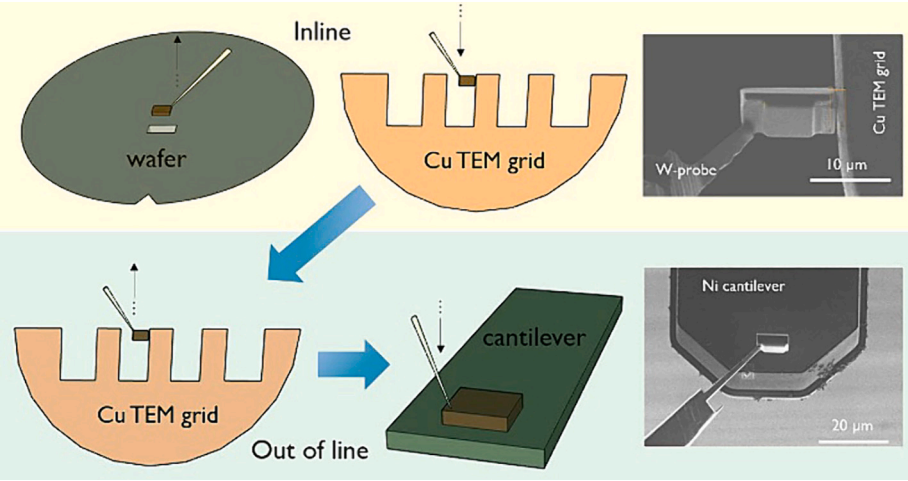

Fig. 6. General principle of full wafer size preparation showing distinction between full wafer size (yellow) and small sample size (green) steps. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

bottom surface of the chunk has been optimized. After lift-out of the chunk, we have found that rotating the W-probe between 60° and 65° around its own axis produces the best configuration to flatten the surface completely parallel with the sample surface. Fig. 5c shows this configuration. After initial attachment of the sample to the cantilever, we also added the same all-around Pt encapsulation step to ensure a strong mechanical fixation. Finishing of the sample is done the same way as for the cross-section procedure. Fig. 5d shows the end result. These samples also can be made in about 90 min of preparation time.

4. Expansion to full wafer size sample preparation

Until now, these preparation methods have been performed only on small pieces of dies in smaller lab-based FIB-tools. Since the nano-electronics industry relies mainly on wafer substrate processing and analysis work inside cleanroom production environments, we expanded our FIB preparation methodology in such a way that RTS SPM samples can be directly retrieved from a full wafer inside the cleanroom during the fabrication workflow.

A challenge which we had to overcome for this was that the existing full wafer size dual-beam FIB-tools are optimized for TEM sample preparation and do not allow for loading tipless cantilevers. Therefore, we combined principles from the first and second approach to overcome this problem. Fig. 6 illustrates that a standard Cu TEM grid is utilized as a transfer medium between the full wafer size (workflow part shown in yellow color in Fig. 6) and small sample size (shown in green color in Fig. 6) FIB-tools.

Chunk extraction is performed in the same way as described in the first approach for the cross-section sample preparation but using a full wafer size Helios 1200 system from Thermofisher. Instead of mounting the sample directly onto a tipless cantilever, it is first fixed on the sidewall of a Cu TEM grid. Once the sample is attached to the TEM grid, the grid is unloaded and is transferred to the small sample size FIB system. This method can be applied both for cross-section and top view sample preparation. Once the TEM grid has been transferred to the small sample size tool, a tipless cantilever is attached again to the FIB sample holder and the procedure proceeds in the same way as described before, i.e. transferring the chunk from the grid to the cantilever, fixating it with Pt and thinning it down until the desired position is reached.

5. Evaluation in RTS SPM measurements

ESPM measurements in RTS mode were carried out to validate the functionality of the prepared samples and to judge their performance

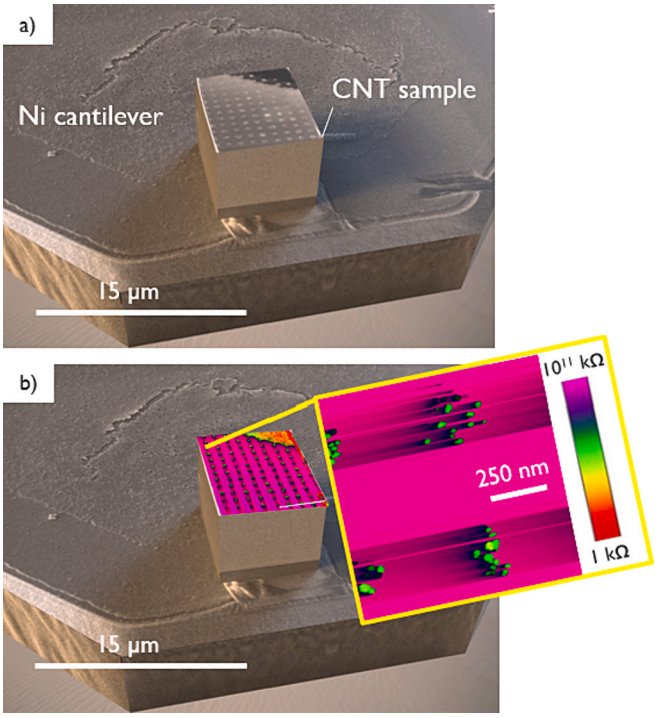

Fig. 7. a) SEM image of RTS SPM MWCNT sample and b) overlay with SSRM results.

compared to the standard mode. All measurements were done with inhouse fabricated boron-doped diamond tip arrays [11,13] using a Bruker Dimension Icon-PT AFM system placed in an Ar-filled glovebox enclosure and equipped with a dedicated C-AFM or scanning spreading resistance microscopy (SSRM) application module. When making contact between the diamond tip and the sample, an electrical current flows from the tip through the device structure over the Pt FIB backcontact into the metallic cantilever and from there further to the current amplifier. Fig. 7 shows a sample consisting of 8–25 nm wide multi-wall carbon nanotube (MWCNT) interconnects integrated into 300 nm diameter and 750 nm pitch contact holes [14,15] which was prepared by the first, combined FIB/epoxy, approach and was measured by SSRM using an applied bias voltage of 500 mV. Note that for better visualization we established in Fig. 7a a 3D model of the cantilever-sample

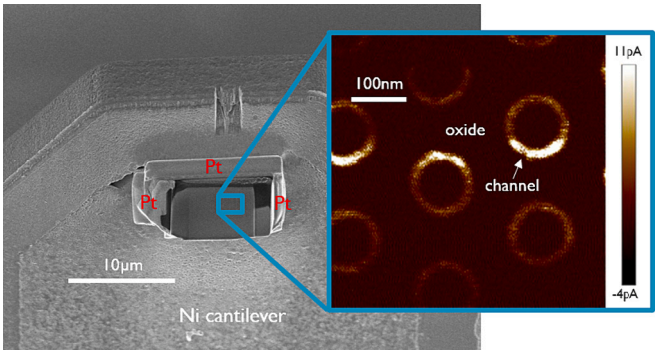

Fig. 8. SEM image and RTS C-AFM measurements of top view sample.

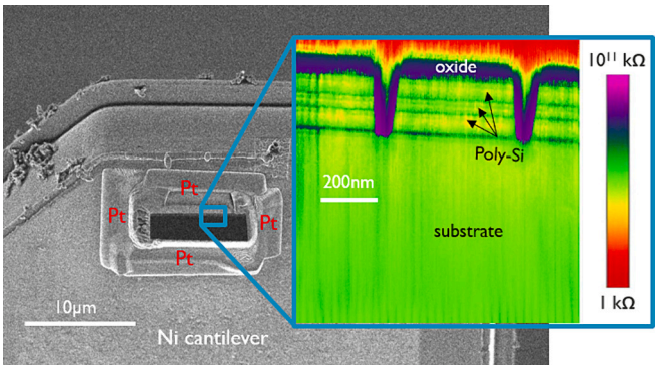

Fig. 9. SEM image and RTS SSRM measurement of inline prepared cross-section sample.

assembly (epoxy glue is not shown) and applied the corresponding SEM and SSRM images as overlays onto it. As can be seen clearly from Fig. 7b, the individual MWCNT structures (green color) surrounded by oxide (purple color) are very well detected with high resolution which proves that the sample is electrically well connected to the cantilever.

Fig. 8 shows a SEM image of a finished top view sample made with the second, FIB-only, approach, together with a corresponding C-AFM measurement. For this measurement, a bias voltage of 6 V is applied. The sample contains memoryhole structures which are 120 nm in diameter, contain a 12 nm thick conductive poly-Si ring channel (bright color) and are embedded in an oxide matrix (dark brown color). The SEM image shows the all-around Pt encapsulation well. Differences in contrast in the channels, measured by C-AFM, indicate preferential conductive paths which gives information about the grain structure of the poly-Si (white regions). All features can be distinguished at high resolution.

Fig. 9 gives an example of an RTS SSRM measurement performed on a cross-section memoryhole sample which is prepared from a full size wafer flow with the second, FIB-only, approach. For this measurement, a bias voltage of 1 V is applied. Here, no conductive channel is present, but three poly-Si gates, divided by thinner oxide layers in between the etched holes are clearly detected due to the difference in electrical conductivity between the poly-Si and the oxide layers. Again, all features can be distinguished at high resolution.

6. Conclusions

This work presents first workflows for the reliable preparation of RTS SPM samples using a dual-beam FIB/SEM system. Hereby we leverage our existing knowhow on TEM specimen preparation to achieve both a high quality and an optimal time efficiency in our procedures. The first approach, based on a combined FIB/epoxy procedure, works well but is more time consuming (due to the intermediate Cu TEM grid fixation) and requires an extra epoxy cementation step to achieve a high mechanical strength of the sample to cantilever connection.

In the second approach, based on a FIB-only procedure, we are able to completely anchor the sample to the cantilever by using an all-around Pt deposition step, resulting in a high mechanical strength. The total preparation time for the FIB-only approach is reduced from about 120 to 90 min.

Furthermore, we expanded the procedures to the preparation of full wafer size samples without having to break the wafers or the need to remove them from their cleanroom environment. Our work demonstrates that it is possible to mount cross-section samples as well as top view samples on tipless cantilevers for both small sample size and full wafer size procedures. The evaluation of the prepared samples in electrical RTS SPM measurements shows that the samples are mechanically stable and electrically well connected to the cantilever. Note that an additional FIB cleaning step might also still be applied after the sample cross-section has already been scanned. In this way the sample surface can be refreshed with minimal efforts. These aspects provide the foundation for the successful characterization of integrated nanoelectronics device structures using the RTS SPM methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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