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Chapter 3

Implementation of MEMS STM scanners

In this chapter, a finite-element-based design study is described of high-speed MEMS STM-scanners. This is followed by an extended discussion of the three main challenges that arise when a MEMS scanner is integrated into an STM setup, namely tip deposition, capacitive coupling between the MEMS scanner and the sample bias / tunneling current, and the mechanical integration of a small MEMS-device in a macroscopic STM system. We start with the design criteria of the MEMS z-actuator.

3.1 Design criteria of MEMS scanners

The design criteria of a MEMS scanner can be divided into two categories: high-speed STM functionality requirements and STM-applicability requirements. The first implies that the moving part of the mechanical structure, in this case the MEMS scanning membrane, should have a high resonance frequency. The minimum resonance frequency of a MEMS scanner, required to achieve a certain imaging rate, is discussed in section 2.8. We see that the MEMS scanner should have a resonance frequency of a few hundred kHz to a MHz. In addition to the high resonance frequencies, the scanners should still have a reasonable scan range. On a very flat and fully periodic surface, a scan range of only a few nm may be sufficient. However, in order to image more typical surfaces with irregularities such as steps and kinks, large-scale periods, domain boundaries, islands and holes, etcetera, a scan range is desirable of at least several hundred nm. Therefore, we will start with MEMS devices that have a maximum actuation range of 600 nm (the maximum membrane displacement before a pull-in event occurs, see section 2.1.2). From figure 2.8, we have seen that there is a trade-off between resonance frequency and scan range. We use finite-element simulations to optimise the MEMS design for both resonance frequency and scan range. Ideally, the MEMS scanner would provide all three (x,y,z) or at least the two fast (x,z) scanning directions. Unfortunately adding extra actuation directions to the MEMS scanner
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usually comes with a lower resonance frequency or with a very limited scan range. Our finite-element-based design of one-dimensional MEMS scanners is discussed in the next section, 3.2. Two- and three-dimensional scanners were also considered during the design process, albeit not to the same extent as one-dimensional scanners. These are discussed in appendix A.

The STM-applicability requirements concern the properties that a MEMS scanner should have in order to be used in actual STM experiments. It is important to keep in mind that a versatile, high-speed MEMS scanner should truly add to the quality of the STM: it should be user-friendly and easily replaceable (much like commercially available AFM cantilevers). The MEMS scanner should not add restrictions or difficulties to the experiments for which it is used and it should deliver high-quality images. To do so, the MEMS scanner should be sensitive enough to be actuated with atomic-scale precision. For the scanners that we have used, the scan range and actuation voltage are comparable to the scan range and actuation voltage of a piezo element (600 nm for the MEMS, at a maximum of +150 V, versus a few µm for the piezo, at a maximum of ±200 V, set by the electronics used). Therefore we expect the sensitivity of the MEMS scanners to be good enough to allow imaging with atomic resolution. The actuation behaviour of the MEMS scanner is discussed further in section 3.3.

When designing a MEMS-based STM, one has to make a choice whether to incorporate it as a tip holder, or as a sample carrier. Using the MEMS as a sample carrier would place serious restrictions on the sample size, weight and preparation procedures. Therefore we have chosen to incorporate the MEMS scanner as the tip holder. Since the size of a MEMS differs so much from the size of a piezo element, regular PtIr or W STM tips cannot be used. A deposition technique should be found which enables the growth of conducting tips on a controlled place on the scanner. The tip requirements, deposition techniques and tip performance are discussed in section 3.4.

The final two integration requirements concern the special geometry of the MEMS scanner. Because the MEMS scanner has a surface overlap with the sample equal to the size of the scanning membrane in our designs (typically 40 µm × 40 µm) the capacitive coupling between the MEMS scanner and the sample will be higher than the capacitive coupling found in a piezo-based STM. This can cause imaging problems, especially during high-speed actuation. This is discussed in section 3.5. Finally, a MEMS scanner has to be fitted onto a piezo element which provides the x,y-scanning (in the case of a one-dimensional MEMS z-actuator) and, optionally, long-range z-adjustment. The large surface overlap between the MEMS scanner and the sample poses
3.2 Finite-element based MEMS scanner design

To optimise our MEMS design process we have modeled the new MEMS structures with the COMSOL finite-element analysis package [88]. In this way, we can model both the actuation properties and the resonance frequencies of our devices. An example of the simulation of a MEMS actuation is shown in figure 3.1 for a scanner with a membrane of 40 $\mu$m x 40 $\mu$m and with legs of 80 $\mu$m length (the legs consist of two parts of 40 $\mu$m each) and 4 $\mu$m width. We have simulated MEMS actuation at both 50 V and 150 V and it is shown that at 150 V actuation voltage, the membrane is displaced inward over 850 nm.1 This MEMS has a fundamental resonance frequency of 261 kHz (figure 3.2). The actuation range of a smaller MEMS scanner (20 $\mu$m x 20 $\mu$m membrane, legs of 40 $\mu$m total length and 2 $\mu$m width), is significantly smaller, namely 60 nm at 150 V actuation voltage (figure 3.3), while the fundamental resonance frequency of this smaller device is as high as 945 kHz (figure 3.4). Thus, for scanning very fast on a smooth single-crystal surface, the smaller MEMS scanner could be used, whilst scanning samples with a rougher surface would require the larger scanner.

Another possible MEMS scanner is a round scanner as shown in figure 3.5. The resonance frequency of this scanner can be tuned by the number and dimensions of the support legs. For a “round” scanner with a thickness of 2 $\mu$m, a membrane with a diameter of 100 $\mu$m, and legs with dimensions 40 $\mu$m x 10 $\mu$m the natural resonance frequency is 720 kHz.

3.3 MEMS actuation tested by AFM and white-light interferometry

Prior to placing the MEMS z-actuators in the STM setup, we first characterised their behaviour by three different methods. We performed these tests for two different scanners: a “fly-swatter” scanner and a “regular” square

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1In reality, the MEMS will travel 1/3 of the gap before collapsing on the actuation plate, i.e. the maximum scan range will be 660 nm. The FEM model takes into account a load on the membrane, derived from the voltage and calculates the displacement directly from this load. As a result of this, it misses the instability.
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Figure 3.1: Static deformations of a 2 \( \mu \text{m} \) thick MEMS scanner with a 40 \( \mu \text{m} \times 40 \mu \text{m} \) membrane, 80 \( \mu \text{m} \) legs (two parts of 40 \( \mu \text{m} \) length) with a width of 4 \( \mu \text{m} \), with 50 V actuation (left) and 150 V (right). Between 0 and 50 V this structure moves over 95 nm; at 150 V actuation voltage, the structure has moved 850 nm. The deformations have been obtained from finite-element calculations, as explained in the text. The colour scale indicates the deformation in the z-direction, normal to the central membrane.

Figure 3.2: Dynamic deformation patterns for the first six resonances of a 2 \( \mu \text{m} \) thick MEMS scanner with a 40 \( \mu \text{m} \times 40 \mu \text{m} \) membrane, legs with a length of 40 \( \mu \text{m} \) and a width of 4 \( \mu \text{m} \). The colour scale indicates the total displacement of the membrane, independent of direction. From top left to bottom right: the first resonance is at a frequency of 261 kHz, the second and third at approximately 600 kHz, the fourth and fifth are at 921 kHz and the sixth resonance frequency is at 1.1 MHz. The deformations and frequencies have been obtained from finite-element calculations, as explained in the text.
3.3 MEMS actuation tested by AFM and white-light interferometry

![MEMS scanner actuated at 150 V](image)

**Figure 3.3:** Static deformations of a 2 µm thick MEMS scanner with a 20 µm x 20 µm membrane, 40 µm legs (two parts of 20 µm length) with a width of 2 µm, at 150 V actuation. Between 0 and 150 V this structure moves over 60 nm. The deformations have been obtained from finite-element calculations, as explained in the text. The colour scale indicates the deformation in the z-direction, normal to the central membrane.

![Dynamic deformation patterns for the first six resonances](image)

**Figure 3.4:** Dynamic deformation patterns for the first six resonances of a 2 µm thick MEMS scanner with a 20 µm x 20 µm membrane, legs with a length of 20 µm and a width of 2 µm. The colour scale indicates the total displacement of the membrane, independent of direction. From top left to bottom right: the first resonance is at a frequency of 945 kHz, the second and third at 1.8 MHz, the fourth and fifth are at 2.3 MHz and the sixth is at 4.1 MHz. The deformations and frequencies have been obtained from finite-element calculations, as explained in the text.
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Figure 3.5: Resonance frequency of a “round” MEMS scanner. The first resonance frequency is found at 720 kHz.

scanner, equipped with an on-chip shielding electrode (the importance of this shielding electrode is explained in 3.5). These scanners are shown in figure 3.6. The “fly-swatter” scanner has been made with the SOI MUMPS process and thus has a thickness of 10 $\mu m$. The out-of-plane stiffness of this scanner is therefore higher than that of a similar structure made with the PolyMUMPS process, with a thickness of 2 $\mu m$. The scanning membrane has dimensions $32 \times 32 \mu m$ and the separation between the moving plate and the silicon substrate is 1 $\mu m$. We have measured the resonance frequency of this scanner using laser interferometry. We start with the intensity profile as shown on the left in figure 3.7. The scanner is actuated at a fixed voltage with increasing frequency. It is not possible to follow the high-speed motion of the MEMS directly with a camera. However, it is possible to see the effect of the resonance of the MEMS motion. This is done by calculating the amplitude ratio between the dark and bright rings. If the MEMS is actuated at a low AC voltage, the rings will move less than their mutual spacing and even with the slow response of the camera, we still see distinct bright and dark areas. It is possible to calculate the difference between these areas, and obtain a significant standard deviation of the intensity from the average value. As the MEMS scanner hits the resonance frequency, the displacement increases, causing the rings to displace more than their mutual separation. Since the camera is too slow to capture this high-speed motion, we only see this as a blurring of the rings. The clear difference between dark rings and light rings is thereby reduced, and the standard deviation decreases. From the plot of the standard deviation versus the actuation frequency, the resonance frequency of the MEMS scanner is determined to be 968 kHz.

In the second test, the MEMS scanner was mounted on the sample stage of an Easyscan AFM [89]. While scanning with the AFM, we actuated the
3.3 MEMS actuation tested by AFM and white-light interferometry

Figure 3.6: Left: Fly swatter scanners, with scanning membrane (1) and supports (2), made in the SOIMUMPS process. The holes are not only etch holes but also intended to reduce the weight of the scanner and thereby increase the resonance frequency. In addition, they will reduce the squeeze film damping of the device and thereby increase the Q-factor. Right: Spider-type scanner with shield, made with the PolyMUMPS process. The scanner consists of (1) a membrane, supporting springs (2), fixed at the supports (3), an actuation plate (4) hidden below the membrane and a layer which shields the actuation plate voltage, of which the edge is outlined in red (5).

Figure 3.7: Left: Interference profile as measured with laser interferometry on the fly swatter scanner. When hitting the resonance frequency, the displacement of the membrane increases, reducing the contrast between the light and dark parts of the interference profile. Right: Experimentally determined resonance frequency of a fly swatter scanner: the resonance frequency was found to be 968 kHz. The standard deviation of the interference profile (left panel) is plotted. As the contrast between dark and light rings disappears when hitting the resonance frequency, the standard deviation decreases.
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Figure 3.8: The actuation of a MEMS device observed with an EasyScan AFM. The apparent height recorded by the AFM switches in accordance with the actuation signal. Left: The actuation of a “fly-swatter” MEMS scanner (left panel of figure 3.6), that was actuated with a 30 V ptp block wave at 0.1 Hz. Right: The actuation of a shielded scanner (right panel of figure 3.6), actuated with a 15 V peak-to-peak block wave at 0.1 Hz.

MEMS scanner with a block wave. Figure 3.8 shows AFM images with a clear distinction between the MEMS z-actuator being in the “up” and “down” positions. This test confirmed the proper up-down motion of the scanner, and its recovery to the “up” position.

To quantify the motion of the membrane between first actuation and collapse, we actuate the MEMS while observing it with a white-light interferometer, which splits a white-light beam, consisting of multiple frequencies, in a reference part and a part directed to the sample. Then, the resulting interference profile of the reflected beam with the reference part is measured, and constructive interference is observed only when the two paths are of exactly the same length. By scanning the beam generator over a preset height range, the topography of the sample can be imaged. In this way, accurate height profiles of the MEMS devices could be determined, as is shown for the shielded scanner in figure 3.9. The motion of the membrane can be calculated by averaging the displacement over a defined area of the membrane and correcting this for the (drift) motion of the fixed parts of the scanner. This results in a displacement curve as shown in figure 3.10. We see that the membrane moved as expected: it could be actuated over distances up to approximately 600 nm for actuation voltages up to 80 V, after which it collapsed, as predicted [51]. The standard deviation in the measured heights was 20 nm because of the
3.3 MEMS actuation tested by AFM and white-light interferometry

Figure 3.9: Height profile of the shielded MEMS scanner of figure 3.8, observed with white-light interferometry. In the left panel, the scanner is shown at 0 V actuation, in the right panel, at 95 V actuation. At the higher voltage, the central membrane is pulled inwards (darker colour).

Figure 3.10: The position of the central membrane of the shielded scanner, measured by white-light interferometry, as a function of the actuation voltage. We see that the scanner moves over approximately 1/3 of the gap before collapsing, as is explained by the theory [51].
resolution limitation of the interferometer. We have measured the heights on several locations of the membrane and find that the entire membrane is actuated evenly to within our measurement accuracy, i.e. 20 nm. This gives us complete freedom in choosing the location of the tip, which will be discussed in section 3.4.

For a fly swatter-like structure, made with the PolyMUMPS process, we find that the membrane collapses onto the wafer surface already at an actuation voltage of 20 V, after which the scanner remains in the ’down’-position. This indicates that at 20 V actuation, the scanner has travelled 1/3 of the gap, being 2 µm in this case. From this, we find that the displacement of the scanner will be 1 nm at 0.3 V actuation voltage. Because the displacement as a function of actuation voltage of the spider-type scanner is more similar to piezo element actuation, and will therefore be more suitable for our electronics, we choose to use the spider-type MEMS scanner for further experiments.
3.4 Tip growth on MEMS

Since the MEMS scanner will function as the tip holder in the STM setup, a process is required to equip the MEMS actuator with a sharp, conducting tip. The tip should be of high quality in order to enable constant imaging at atomic resolution, without tip changes during the measurement. The tip requirements will be discussed in section 3.4.1. The difficulty for tips on MEMS is the small size of the MEMS scanner. Most other STM systems, based on piezo elements, have some form of tip holder, such as a capillary or a clip. The tip is usually made out of a W or PtIr wire with a diameter in the order of 100 µm or more. This is already larger than our entire MEMS scanner, which is a square with sides of only 40 µm. Also the fragility of MEMS structures would make it very challenging to mount a wire-based tip on a MEMS device. Therefore, we have explored the possibility to fabricate a tip-like structure on top of the MEMS device by direct deposition. An additional problem in the implementation of an STM tip in a MEMS might arise from the native oxide present on the MEMS surface, which will most likely also be present between a deposited or grown tip and the MEMS scanner. Such an oxide may seriously limit the conductance between the tip and the MEMS device.

The difficulties encountered in the fabrication of suitable tips on a MEMS scanner might be one of the main reasons why previous research on MEMS STM/AFM papers has not led to a wider use of these devices. In most MEMS STM/AFM papers, very limited information is provided about tip deposition or growth. In fact, many papers present only SEM images of the MEMS device and no STM or AFM images obtained with the MEMS scanners. In the few papers that do present STM or AFM images, the resolution is rather modest. Usually, images are shown of calibration gratings with feature sizes of tens to hundreds of nm. The tip requirements are considered below. Then, a short overview of possible deposition methods will be given, followed by a discussion of the selected deposition technique and the tip quality obtained with it.

3.4.1 Tip requirements

A tip that is compatible with our MEMS scanners and allows imaging with atomic resolution in Scanning Tunneling Microscopy experiments, must have the following properties:

1. Sufficiently high electrical conductivity, combined with a good contact to the MEMS scanning membrane through the silicon native oxide
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... and absence of other insulating surface layers. This is especially important for the tip apex.

2. A sufficient tip length is required to ensure that the tip is the first element to make contact with the sample, rather than other components of the scanner. The ratios between the dimensions of the tip, sample and scanner are very different in a MEMS scanner than in a piezo-based scanner. The MEMS scanner is on a die of typically a few mm in size. Due to the small size of the MEMS, the tip cannot be longer than several tens of micrometers. If the sample and scanner are not aligned well enough, chances are high that the MEMS scanner or further parts of the wafer will crash into the sample before the tip comes into tunneling range. The combined height of the silicon layers of the MEMS scanner is 5 µm. If the scanner is on a 2 mm diameter wafer, approaching a 5 mm diameter sample, for a 1 µm long tip on the 5 µm thick substrate the maximum allowable alignment error is 0.34°. For a 10 µm tip, the allowable error is 0, 86°.

3. The sharpness of the tips apex is important to routinely obtain atomic resolution. We aim at an end radius of 10 nm.

4. The resonance frequency of the tip structure should be sufficiently high, i.e. > 1MHz, in order to avoid image distortions due to scanning-induced tip deformations.

5. The production process should be reliable: it should enable us to obtain a fully reproducible result so that we can compare results for different scanner designs without too much influence from differences between tips. For the first tests, ease and speed of tip production are not yet a criterion. These considerations may become relevant in a later stage.

6. The tip deposition has to be performed below 300°C, to prevent diffusion of gold atoms of the contact pads into the silicon structure.

3.4.2 Deposition techniques

To identify candidate techniques, we have first conducted a literature study of various etching, deposition, and growth techniques [90]. Any etching method applied after completion of the MEMS device would severely affect its mechanical properties. Tip etching can be considered only if it can be combined
3.4 Tip growth on MEMS

with one of the etching steps of the scanner (i.e. during the lithographic process used to create it). However, in the commercial process that we use for MEMS fabrication [56], there is no room for a tip deposition step, and therefore tip production methods that involve etching could not be considered. As a second option, we have studied the growth of silicon whiskers by a VLS (vapour-liquid-solid) growth mechanism as a technique to deposit a tip on a MEMS scanner. VLS is a widely studied technique and the reliable growth of nanowires is an active field of research. The vapour-liquid solid growth mechanism was first proposed by Wagner and Ellis in 1964 [91]. Since then, there have many reports on nanowire growth techniques. Some of them are about the growth of semiconductor nanowires on Si, for instance, [92–98]. Others are about the growth of silicon nanowires, for example [99–103]. After a short study, we were able to deposit pillar-like structures on the polycrystalline silicon surfaces of our MEMS scanners by VLS, a typical being result shown in figure 3.11. Unfortunately, we could not gain sufficient control over the tip length, orientation, apex sharpness or deposition location to the extent that would be necessary to produce reproducible STM tips on our MEMS scanners without putting in a more extended research effort. Therefore, we have turned our attention to a third technique. Electron-Beam Induced Deposition (EBID) is explained schematically in figure 3.12. The MEMS structure is placed in a Scanning Electron Microscope (SEM). A precursor gas containing the desired growth material (in our case, Pt) is admitted into the vacuum of the SEM near the MEMS device. Figure 3.13 shows an SEM image of a MEMS structure together with the gas injection needle. The electron beam is focused at the location where tip growth is desired. The precursor gas decomposes under the influence of the electron beam and the platinum atoms remain on the surface while the other components of the gas desorb. In this way, the process results in the formation of a pillar of several micrometers height in a time frame of several minutes up to one hour, depending on the precursor gas used and the parameters of the electron beam. For an extensive discussion of the theory behind EBID, we refer the reader to [104]. We have investigated the best way of growing EBID tips and have tested the conductance of these tips [105], as is discussed in sections 3.4.3 and 3.4.4.

3.4.3 Platinum STM tips by EBID

For the deposition of platinum, we have used two different precursor gasses: $(CH_3)Pt(CpCH_3)$ (Methylcyclopentadienyl platinum trimethyl) and $Pt(PF_3)_4$. 

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Figure 3.11: Left: SEM micrograph taken at 0° (i.e. perpendicular to the surface) of various VLS deposited structures on silicon, scattered over the surface. None of the tips are perpendicular to the silicon surface. Right: SEM micrograph of two VLS tips, taken at an angle of 45° to the silicon surface. We see that these tips are standing on the surface, albeit not completely upright. The tips have completely different apexes, while they are grown simultaneously. In the top left corner, two more tips can be seen, lying on the surface.

The latter is also known as tetrakis. There are many reports on EBID growth with these precursors and different purities have been reported for deposits obtained with both precursors, as has been summarised in [104, 106]. For the \((CH_3)Pt(CH_3)\) precursor, purities of 10 to 15 at.% Pt are reported before annealing with the main contaminant being carbon: the structures have a carbon content of 65 to 81 at.% depending on the deposition parameters and post-treatment methods used [107]. For the tetrakis precursor, which is carbon-free, a composition has been reported of 58 at.% Pt, 32 at.% P and 10 at.% F with no carbon contamination [108] with the highest purity reported for gas-phase platinum deposition being 83 at.%. For liquid-phase platinum EBID, a purity of 90 at.% Pt is reported [109]. Liquid-phase EBID introduces the necessity to shield the liquid from the vacuum environment of the SEM. To be able to do so, our MEMS scanner would have to be sealed, with a liquid inside on top of the scanner to allow deposition from the liquid on the scanning membrane. Because our MEMS scanner will be used as a scanning probe microscope, it would have to be unpacked after the deposition. A sealing and subsequent unpacking step would be a high-risk procedure for such a fragile structure. Moreover, after unpacking the MEMS would have to be placed in a critical point dryer immediately to prevent sticking of the membrane to the actuator plate after evaporation of part of the liquid. Con-
3.4 Tip growth on MEMS

Figure 3.12: Schematic overview of the EBID deposition process. The silicon MEMS scanner is placed inside an SEM. A Gas Injection System is inserted above the scanner to let in a precursor gas. The electron beam is focused at the desired tip location. Under the influence of the electron beam, the precursor gas is decomposed. The metal atoms, in this case platinum, stick to the surface and other molecules fly off. At the point where the platinum is deposited, a column is formed with a length of several micrometers in several minutes to one hour.

Figure 3.13: SEM micrograph of a wafer carrying several MEMS scanners, with the GIS (gas injection system) needle positioned above it, to allow inlet of precursor molecules right above the sample.
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sequently, liquid-phase EBID would be too complicated to regularly deposit tips on our different MEMS-scanners.

As explained in section 3.4.1 we want to deposit a sufficiently long tip without giving up the high resonance frequency or stability of the structure. With EBID, it is possible to create a base structure by scanning the electron beam in a pre-defined pattern over the surface before focusing on one spot to create the sharp tip. The result of such a procedure is shown in figure 3.14. This structure was grown fully with the \((CH_3)Pt(CpCH_3)\) precursor in three stages, beginning with scanning the electron beam over a square on the surface to make the support, then scanning in a circular pattern to grow a pillar with a diameter of 0.5 \(\mu\)m, and finally focusing the electron beam at one position to grow the sharp tip. Because the tetrakis precursor results in deposits with a higher platinum content, we have also tried to grow this type of structures fully with the tetrakis precursor. This turned out to be an extremely unreliable method of creating structures, probably due to a high mobility of the atoms during the deposition. A typical result is shown in figure 3.14. In addition, the growth of larger structures takes a much longer time with tetrakis than growth with \((CH_3)Pt(CpCH_3)\) (for a pillar of 5 \(\mu\)m, the respective growth times can be as much as 1 hour vs. 10 minutes). This poses a risk of quickly running out of the tetrakis precursor, which is costly and takes time to replace.

The sharp end of the tip is built up, ideally, from just a few atoms. Thus, the conductance of the tip as a whole depends heavily on the composition of the apex. As explained above, to decrease the probability that one of the edges of the scanner reaches the sample before the tip, a long tip is required. For this purpose, we have been forced to sacrifice some of the conductance of the base of the tip. We have therefore chosen to grow two-stage tips where the larger base structure is grown with the \((CH_3)Pt(CpCH_3)\) precursor and on top of this, a tetrakis tip is deposited. This turns out to give reliable tips with sufficient length; tips up to 18 \(\mu\)m have been grown. A typical result is shown in the left panel of figure 3.15.

It should be noted that the EBID process is not 100% reliable. After the gas injection needle is inserted, the SEM system exhibits significant drift. If the tip growth is started before the system is has stabilised again, there is a strong tendency to grow side-tips, of which an example is shown in the right panel of figure 3.15. This tip was deposited in exactly the same circumstances and only minutes before the tip shown in the left panel of the same figure. At other occasions, multiple tips were grown simultaneously, as is shown in figure 3.16. These side-tips, multiple tips and other deformations are probably all

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Figure 3.14: Left: Large EBID structure consisting of a base with sides of 1 µm with a pillar, ending in a sharp tip (apex diameter approximately 25 nm) grown with the (CH₃)Pt(C₆H₅) precursor in 13 minutes. The length of the structure is 9.1 µm. Right: A typical result of a larger structure grown with the tetrakis precursor. The base was set to be 1 µm. The diameter of the tip apex is approximately 20 nm. Attempted growth of larger structures with the tetrakis precursor gives very unpredictable results. If a tip is deposited on top of a larger structure, these underlying structures usually change shape. The base structure of this tip was grown in 1 hour, the tip in a few minutes.

related to residual drifting of the SEM.

3.4.4 Conductance measurements of EBID tips

Many experiments with EBID have been done depositing in-plane structures and the composition and conductance of these structures has been tested [104, 107, 110–118]. It has been found that the composition of this type of structure is far from ideal. Of the two precursors that we have used, the tetrakis precursor gives the best result, being 48 at. % Pt when deposited at room temperature [107]. This means that there is a considerable amount of other atoms present and the exact composition of the STM tips that are grown by EBID is unknown, which is a problem especially at the tip apex, where the structure consists of only a few (hopefully metallic) atoms. To test the functionality of platinum EBID tips in scanning tunneling microscopy, we have performed two experiments: one where the EBID tip was incorporated in an electrical circuit to directly measure its conductance, and another where the EBID tip was integrated in a known STM system, to test its functionality under tunneling conditions.
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Figure 3.15: Two tips grown by deposition of a platinum base structure from $(\text{CH}_3)\text{Pt}(\text{C}_6\text{H}_5)\text{CH}_3$ followed by the deposition of a tip via the tetrakis precursor. These tips were made under the same circumstances and with the same deposition conditions. The base layer of this tip was grown in 90 minutes, followed by the tip deposition which took 30 minutes.

Figure 3.16: MEMS scanner equipped with EBID tip. Several tips have grown simultaneously, probably due to residual drifting of the SEM.
Direct conductance measurement

In the first experiment, we have tested the conductance by making a direct contact in the SEM between the tip and the metal-wire tip on a separate nanomanipulator [119]. For this experiment, we first deposited an EBID tip on the MEMS substrate. The nanomanipulator was equipped with a tungsten needle and placed in the SEM. While using the SEM to look at the MEMS tip and the nanomanipulator needle (as is shown in figure 3.17), we could position the nanomanipulator needle closer to the EBID tip and establish a mechanical contact. There were a few factors in this experiment that complicated an accurate measurement of the conductance. While approaching towards the EBID tip with the nanomanipulator needle, there was no information on their relative height and thus we did not know precisely at what height the EBID tip was contacted. In addition, the tungsten needle was probably oxidized and because we did not have a cleaning method available in the SEM, this oxide skin probably has added to the resistance of the tip-tip contact. Also, the carbon deposition which takes place while prospecting with the electron beam probably has contaminated the contact and increased the resistance. Still, we expect that a meaningful upper estimate can be made in this way of the resistance of the EBID tips. First, a reference measurement was carried out of the resistance of a MEMS device by itself, i.e. without EBID tip present. We find the upper limit of the resistance to be 0.7 MΩ. The sheet resistance of the Poly1 silicon layer is known to be 1-20 Ω/sq [57]. From the geometry of the scanners, we expect the resistance from the contact pads to the actuation pad to be between 60 Ω and 1.2 kΩ, while the resistance from the contact pads to the tip position on the scanning membrane is expected to be between 25 Ω and 500 Ω. The huge difference between these low numbers and the high upper estimate of 0.7 MΩ has can explained by the two complications mentioned above. In addition, as was mentioned at the beginning of section 3.4, the electrical contact between Pt EBID tip and the MEMS surface was limited due to the native oxide on the MEMS surface. We find that the resistance of the tetrakis tips varies greatly between, and even during, measurements. Typical results are shown in figure 3.18 for two different EBID Pt tips. For both measurements, it was not possible to determine the height at which the tip was contacted. The lowest obtained value for the tip resistance was 0.70 ± 0.34 MΩ, which is as low as the resistance found when contacting the MEMS device directly at its surface, i.e. without tip present. Even though the resistance values found here indicate that the electrical connection between the tip apex and the contact pad is far from perfect, these
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Figure 3.17: The process of contacting an EBID tip with a W-tip on a nanomanipulator. With an external control system, the W-tip (1) can be positioned in three directions. To establish a contact with the EBID tip (2), the nanomanipulator tip is moved in-plane towards the EBID tip. If it is found that the nanomanipulator tip is positioned too high to establish a contact, it is lowered and moved towards the EBID tip again.

Figure 3.18: Tip resistance, measured with a nanomanipulator contact on a Pt tip grown by EBID from a tetrakis precursor. Left: Between points A and B, the average resistance is 12.0 MΩ. Right: Between points A and B, the average resistance is 0.69 MΩ.

values are still very small in comparison with the high resistance of 1 GΩ typical for the tunneling gap between the tip and the sample in STM. Even if the resistivity of the tip-MEMS combination is fluctuating, this need not be a problem as long as the fluctuations are only a fraction of the tunnel resistance of 1 GΩ. Fluctuations in the MΩ range will not be visible in the tunnel current. In conclusion, the high resistivity of the tips and the fluctuations in measured resistivity between experiments will not render the STM experiment impossible.
3.4 Tip growth on MEMS

EBID tip in tunneling configuration

In the second experiment, we deposited an EBID tip on a tungsten STM tip (shown in the left panel of figure 3.19) which was then placed in the EasyScan STM [89] together with an HOPG sample. This allowed testing of the EBID tip in a known, working system, where the EBID tip was the only new element in the system. We observed that it was possible to establish a tunneling contact between the EBID tip and the sample. To observe the behaviour of the EBID tip in more detail, we measured an I//V spectrum on HOPG, as is shown in the right panel of figure 3.19. The EBID tip shows Ohmic behaviour, even though the I/V curve has an offset which is caused by a voltage offset in the control electronics. Atomic resolution on HOPG was not achieved in this setting. The tunnel resistance in this experiment was approximately 10 MΩ, so a tip resistivity of 1 MΩ would add considerably (10 %) to the total resistance.

3.4.5 Towards atomic resolution

As mentioned above, we have not achieved atomic resolution on HOPG using the tungsten tips equipped with a platinum EBID tip at the apex. Although the EBID tips presented here seem to have an apex of a few nm upon first inspection with the SEM, it is very likely that the last part of the tip, which
Chapter 3 Implementation of MEMS STM scanners

is not visible by SEM inspection, is blunt. To obtain high resolution in STM imaging, it is necessary that the last point of the tip is very sharp; if this is not the case, atomic resolution will not be possible. It is unlikely that the EBID tips have such a sharp apex, considering the contamination from both the precursor gas and from the SEM electron beam, and the dynamics of the growth process. Still, the STM experiment and the conductivity measurement using the nanomanipulator show that the combination of EBID tip and MEMS has a sufficiently high conductivity to allow tunneling experiments, despite the low platinum content of the EBID tip and the native oxide present on the MEMS surface.

Several improvements can be imagined. The first possibility is the EBID deposition of gold tips. It is known that gold tips can be deposited with a higher purity, having an Au atomic content of 80% to even 100% [106, 120]. If the lack of conductance of the Pt EBID tips was caused by the low platinum content, this can be solved by depositing Au tips. Alternatively, better tips might be incorporated on the MEMS by transferring ready-made tips from a wafer to the MEMS. This will be discussed in more detail in section 5.4.1.

A more ideal solution would be to integrate the tip growth with the lithographic process that is used to make the MEMS scanner. This would provide tip deposition directly on the MEMS silicon surface, without oxide layer between the MEMS and tip. For MEMS scanners produced in a custom process, integration of tips during the lithographic process is very much desirable over any deposition method that can be applied afterwards.

3.5 High-speed MEMS scanner actuation and capacitive coupling

In all STM experiments, also with piezo-based STMs, capacitive coupling is a primary source of interference. Parasitic capacitances can be modelled as capacitances coupled directly to the input of the pre-amplifier, adding parasitic currents to the tunneling current. The main source of capacitive coupling is with the actuation voltages on the MEMS scanner. In the geometry of our STM, the tunnel current is measured via the sample, so the important parameter is the capacitive coupling between the MEMS actuation plate and the sample. We suppose an actuation plate-sample separation of 10 µm and a surface overlap of 40 µm × 40 µm. We can estimate this capacitive coupling using equation 2.1 and find:
3.5 High-speed MEMS scanner actuation and capacitive coupling

\[ C = \frac{\epsilon_0 A}{d} = \frac{8.85 \cdot 10^{-12} \cdot 1.6 \cdot 10^{-9}}{10 \cdot 10^{-6}} F = 142fF \]  \hspace{1cm} (3.1)

This calculated capacitive coupling is an upper limit. It assumes that the actuation signal to the MEMS is not shielded from the sample in any way. In reality, the MEMS scanning membrane is set at a (fixed) sample voltage which will act as a shield between the actuation plate and the sample. What is left is the stray capacitance. This value is difficult to calculate directly from the geometrical properties of the scanner. It can be estimated experimentally, when the MEMS scanner is integrated in the STM scanner. The parasitic current running through the STM sample when an AC voltage is applied to the MEMS actuation plate, keeping the MEMS membrane at 0V, can then be measured. In this way, the capacitive coupling has been estimated to be $70\,\text{fF} \pm 6.1\%$. This estimate is a lower limit for the capacitive coupling between the MEMS actuation plate and the sample: it was done before the MEMS and sample were in tunneling range, so in the STM experiment, they will be at a smaller distance to each other. The true capacitive coupling of the MEMS actuation plate and the sample will be between the lower bound of $70\,\text{fF}$ and the upper bound of $142\,\text{fF}$. The parasitic current can be calculated from the capacitive coupling. If we assume an actuation voltage of 1V and an actuation frequency of 10 kHz, using the lower bound (70 fF) of the parasitic coupling, we find the parasitic current to be:

\[ I_p = 2\pi \cdot C \cdot f \cdot V_A = 2\pi \cdot 70 \cdot 10^{-15} \cdot 10 \cdot 10^3 \cdot 1A = 4.4nA \]  \hspace{1cm} (3.2)

The parasitic current will increase linearly with the applied actuation voltage and with the actuation frequency. We find that the lower bound of 70 fF of the capacitive coupling is already much too large to allow scanning even at moderate speeds. Since STM experiments are usually done with a tunneling current in the range of 100 pA to 2 nA, a parasitic current of 4.4 nA at only 10 kHz actuation frequency and 1 V actuation amplitude will inhibit STM experiments. To minimize the capacitive coupling, and thus be able to scan at higher actuation frequencies, we have designed a scanner with on-chip shielding, as is shown in the right panel of figure 3.6 on page 37. The shielding of the membrane lowers the stray capacitance. The shielding is extended to also cover the actuation lines in order to further minimize capacitive coupling between the MEMS die and the sample or other electric components of the STM, such as the electrodes of the xy-scanning piezo element. The capacitive coupling of the shielded scanner to its surroundings has not been measured in practice yet.
3.6 Mechanical integration of a MEMS scanner

Integrating a MEMS scanner into Scanning Tunneling Microscopy requires one to address the problem of aligning the MEMS scanner and the sample. As is explained in section 3.4.1, for MEMS the size ratios between scanning element, tip and sample are completely different from those in piezo-based scanners. Both sample and wafer are millimetre-sized which is very large compared to the MEMS scanner and tip, which are micrometer-sized. For our first MEMS STM experiments, the MEMS wafer was mounted on a piece of printed circuit board (PCB), which was mounted in turn on the piezo element, as shown in figure 3.20. The contacts to the MEMS scanner were established by wire bonding from the PCB to the MEMS scanner. The approach process in this configuration was rather demanding, as can be seen from figure 3.21. The figure illustrates how difficult it is to see whether MEMS and sample are properly positioned and aligned with respect to each other: for clarity, a schematic representation is included. As discussed in section 3.4.1, for a 10 µm long tip the MEMS scanner and sample have to be aligned to within 0.85°. If the alignment error is larger than this value, the wafer edge will touch the sample before a proper tunneling contact is
established between tip and sample. For the fragile MEMS scanner this can be catastrophic, as shown in figure 3.22. Exchanging MEMS scanners is more complicated than exchanging tips and should be avoided. A first, relatively easy way of reducing this problem is to place the MEMS scanner at a corner of the wafer. By slightly tilting the wafer the crash risk is reduced. In addition, it is possible to use smaller wafers, to further reduce the chance of crashing. As explained before, longer tips are also beneficial. However, for further research a dedicated MEMS holder will be necessary.

3.7 Summary

In this chapter, we have shown how finite-element analysis can be used as a tool for the design of MEMS STM scanners, along with a series of tests on two of these scanners to study their functionality as STM devices, before incorporating them in an STM setup.

In section 3.1, the design criteria on high-speed MEMS scanners were discussed. These can be divided into high-speed scanning requirements, being a high resonance frequency combined with a scan range of at least a few hundred nm, and STM applicability requirements, which can be summarised as “user-friendliness”: while MEMS-based STM experiments introduce new, technical challenges, eventually these tests should lead to a MEMS STM scanner that can be used in a wide range of experiments without adding restrictions or
difficulties to the experiment. Because of this, the MEMS scanner should not be used as a sample holder, since this would place severe restrictions on the sample size, mass and preparation procedures. Consequently, the MEMS scanner has to be equipped with a tip.

In section 3.2, we showed how finite-element simulations can be a useful tool in designing MEMS STM scanners. These simulations can be used to tune the geometry of the MEMS scanner to achieve an optimal balance between a high resonance frequency and the minimum scan range required. Also, finite-element analysis can be used to analyse new geometries, such as the “round” scanner (figure 3.5).

After these design considerations, a series of tests was done with two of the MEMS scanners, namely the “fly-swatter” scanner and the “spider” scanner (both shown in figure 3.6). The first tests confirmed that both MEMS scanners indeed have very high resonance frequencies: for example, the resonance frequency of the fly-swatter scanner was found to be 968 kHz. Furthermore when the spider scanner was actuated, the membrane moved down controllably over 1/3 of the gap between membrane and actuator plate before collapsing, as predicted by theory.

Then, the problem of tip deposition on the MEMS scanner was addressed. We have shown that electron-beam induced deposition (EBID) of platinum can be used to deposit tips of a few µm length on the MEMS scanners. From literature [106], it is known that the composition of these platinum EBID
3.7 Summary

structures is not perfect. Rather than being composed of platinum alone, the EBID tips contain up to 81 at. % carbon or, even if a carbon-free precursor is used, the highest purity reported in literature corresponds to 58 at.% Pt, 32 at.% P and 10 at.% F [108].

We have tested the conductivity of the platinum EBID tips with two experiments: a direct conductivity measurement and a performance test under tunneling conditions. Both experiments showed that the combination of a MEMS scanner, with its native oxide and an EBID-deposited platinum tip has a high resistance in these cases up to $0.69 \ M\Omega$. This high resistance is still below 1 % of the resistance of a typical tunnel gap (which is in the order of $1 \ G\Omega$), so we expect that it is possible to perform STM experiments using these EBID tips.

After this, the capacitive coupling of the MEMS scanner to the sample was estimated to be between 70 $fF$ and 142 $fF$. The resulting parasitic current, which will be seen superimposed on the tunnel current signal, was found to be several nA for a MEMS actuation frequency of tens of kHz. This is too high to allow unperturbed STM measurements. This motivated us to use a shielded scanner (shown in the right panel of figure 3.6), in which the actuation lines as well as the actuation plate and most of the membrane and legs are covered with an on-chip shield. This shield will considerably lower the stray capacitance and therefore, this scanner was used for the MEMS STM measurements shown in section 5.2. We have not explicitly measured the stray capacitance of the shielded scanner but did observe that it does not influence the tunnel current.

Finally, the mechanical integration of a MEMS scanner into an STM setup was considered. This integration can be problematic because of the relative sizes of the micrometer-sized MEMS scanner and the millimetre-sized sample. When the MEMS scanner die and the sample are not aligned within less than one degree, there is a high chance of crashing one side of the wafer into the sample before the tip is in tunneling range. We have shown how the MEMS scanner was mounted onto the piezo element for the first experiments. For further development of the MEMS STM scanner, a dedicated MEMS holder would be desirable to improve and standardise the alignment between MEMS scanner and sample.

One aspect that has not been considered here is the maximum scanning speeds of MEMS STM scanners under UHV conditions, as the transition from atmospheric pressure to UHV conditions eliminates the squeeze film damping, drastically increasing the Q factor of the MEMS scanner. Therefore, the MEMS scanners will be more sensitive to vibrations under UHV conditions.