TAILORED SPM PROBES FOR DUAL-TIP FORCE MICROSCOPY

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Abstract

This work reports on two novel SPM methods which both use tailored dual AFM tips. The first method, so called dual-tip magnetic force microscopy (DT-MFM), is based on two-pass scanning with switching nonmagnetic and magnetic tip during the scan. Thanks to segregation of topological and magnetic scans using two different tips this technique reduces invasion of high local magnetization of the tip apex and is suitable for soft magnetic sample. The second method, so called dual-tip force spectroscopy (DT-FS), is based on special dual-probe for bio-applications to study the mechanical properties of cells. It consists of two cantilevers; one having a sharp tip, the other having a spherically blunted tip. The sharp tip is used for topography imaging with high resolution, while blunt tip is used for measuring of force distance curves. The larger area of probe-cell contact results in averaging local variation in the cell rigidity compared to the one measured with a regular sharp probe. The switching between sharp and blunt tip is realized by means of integrated magnetostrictive actuator.

Keywords: AFM probes, dual-tip, magnetic force microscopy, force spectroscopy

1. INTRODUCTION

Since its invention in 1986 [1], the atomic force microscope (AFM) has proved its versatility in various fields of application. Besides its primary function, topography imaging, nowadays we can use the dozens of different scanning modes which we call as a Scanning probe microscopy (SPM). The heart of an SPM consists of a sharp tip placed at the end of a single cantilever. Modifying the tip (chemically or physically) allows various properties of the sample surface to be measured. For this purpose a large variety of commercial tips are offered. In general, each technique uses single probe, i.e. one tip on single cantilever which limits overall performance. In order to enhance speed or versatility of SPM there have been a few reports, on dual/multi cantilevers for various purposes. For example, a dual silicon cantilever device with a bimorph actuator was developed to reduce the tip-wear problem [2]. Similarly, dual cantilever probes were used for in-situ imaging and mechanical operation for cutting of bio-molecules [3, 4]. Many other groups published works on multi-cantilever-probe systems. The parallel arrays has been used for imaging and lithography [5, 6, 7], data storage applications [8, 9], and parallel spectroscopy [10].

This work reports on two novel SPM methods which both use tailored dual AFM tips. The first method was developed to measure magnetically soft samples. The perturbation of stray field of the magnetic tip was minimized. In the second experiment, a special dual-probe was developed to measure high resolution topographies by a sharp tip and mechanical properties by a spherical tip.

2. DUAL-TIP MAGNETIC FORCE MICROSCOPY

2.1. Principle and fabrication of DT-MFM

Standard magnetic force microscopy (MFM) is two-pass method, i.e. within the first step magnetic tip takes topography and next, in the second step, the tip is lifted up to a constant height (~ 20 -100 nm), to lower van der Waals forces and to image magnetic forces. The magnetic contrast is obtained via the evaluation of the phase shift of the vibrating cantilever, and its value corresponds to the tip-sample magnetic interaction [najdi]. The main disadvantage of the standard two-pass magnetic force microscopy (MFM) method is that the magnetic tip is within the first pass in very close contact with the magnetic sample explored. This is not suitable when soft magnetic samples are scanned. To avoid the sample touching, we have developed dual-tip magnetic force microscopy (DT-MFM). Within the method, instead of using one tip for both, topography and magnetic scanning, we use dual-tip with separated functionalities - one is for topography, and the second one for the magnetic scanning. Two tips are in fixed distance and fill separate roles - one is used for the topography only, and the second one for the magnetic-field mapping only. The tips imagine regions distant e.g. from 100 nm to 2 µm - the pitch is defined by the fabrication process. The method is suitable for flat surfaces, i.e. to explore effects in ferromagnetic layers, or in thin magnetic patterned objects. The DT-MFM also is based on a two-pass scanning. The idea of the DT-MFM scanning is explained in the **Figure 1(a)**. Within the first pass the non-magnetic tip Tip1 oscillates at its resonance frequency *f1*, scans the sample-surface in tapping mode and evaluates the topography in the region at the distance of Δx apart from the magnetic tip. In the second pass the magnetic tip Tip2 oscillates at its resonance frequency *f2*, and scans the magnetic field in close proximity to the surface without it's touching. The distance d_m between the magnetic tip and the sample is defined by the dual-tip height difference, and by the sample profile below the Tip1 and Tip2. The distance d_m is defined within the fabrication process can be tuned also by setting the lift distance or by the Tip2 oscillation amplitude.

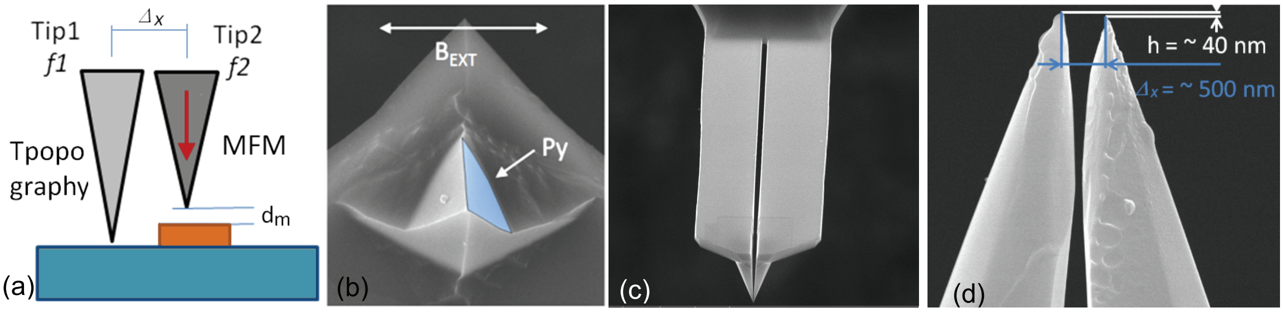

Figure 1 Principle of the dual-tip MFM technique. (a) Topography in the first-pass is performed by nonmagnetic Tip1 at the resonant frequency *f1*, and the phase shift is evaluated by magnetic Tip2 at the resonant frequency *f2*. SEM pictures (b), (c), (d) show the fabrication process of the dual-tip by FIB - (b) tip after Py evaporation onto one of the tips sidewalls (top-view at the end of cantilever), (c) beam and tip after splitting into half using FIB, and (d) is detailed view of the very end of the dual-tip - non-magnetic (left) and magnetic (right)

The dual-tips were prepared as follows. First, magnetic layer (Permalloy ($Ni_{81}Fe_{19}$; Py)) was evaporated onto one sidewall of the tip (commercial tip NSG01) under the specific angle to create Py ferromagnetic triangle (b)), which holds single domain magnetic state during the scanning [11]. Mechanical stress between the Py layer and the tip was partially eliminated by a 5-nm Au seed layer (e-beam evaporation of Au prior to the evaporation of the Py layer). In the next step, focused gallium ion beam microscope (FIB) was used to cut the tip and the beam into two separated tips and beams - one magnetic with the Py triangle prepared as described above, and the non-magnetic one. **Figure 3(c)** shows the overall view onto the final dual-cantilever with the dual-tip located at the end of the beam. The beams shown have resonant frequencies *f1* = 168 kHz and *f2* = 173 kHz, respectively. **Figure 1(d)** shows the detail of the tips apexes. The right tip, the shorter one, is the magnetic one, covered with 15-nm-thick Py layer on its right side. The Py thickness was optimized in our previous work [12]. The left tip is non-magnetic, located 500 nm apart from the magnetic one.

2.2. Results and discussion

As mentioned above, standard MFM technique performed on soft magnetic objects often demonstrate the occurrence of the tip-induced perturbations. Therefore, the functionality of our dual-tip MFM method we have tested by scanning the magnetically soft object, Py submicrometer-sized elliptical object. The 40-nm thick ellipses were fabricated by electron beam lithography (EBL) using PMMA resist, Py deposition and lift-off process. Our tests were realized on the Py ellipse of the dimensions of 400 nm x 800 nm (topography shown in **Figure 2(a)**) with the ground magnetic state with one vortex located in its centre. All MFM experiments were performed by the same scanning system NTegra Prima (produced by NT-MDT co.).

The comparative test started with standard MFM method and commercial tip (MESP from Bruker) at different lift distances and scanning directions. **Figures 2(b), (c)** show typical magnetic images of the ellipse obtained in the scans in the horizontal (b) and the vertical (c) directions. The scans were done at the lift distances 30 nm and 60 nm, respectively. Similarly to Ref. [13], the tip changes the magnetic state of the ellipse during the scanning in both of the scanning direction. Obtained magnetic images are strange, the tip perturbations mix the magnetic states drastically, so one can hardly recognize the ground or other magnetic states of the ellipse. Also, it is impossible to distinguish the influence of individual passes onto the images.

After measurements by standard MFM method we scanned the same Py ellipse by DT-MFM method (**Figures 2(d), (e)**). In both of the **Figures 2(d), (e)** we can see typical magnetic contrast for ellipse with one vortex located in its centre [13] (right part of the figures). In the left part of the images, residual picture of the van der Walls forces from the topography Tip1 is visible - magnetic and van der Walls forces are delocalized. In the first pass the amplitude of the beam 1 was set to 30 nm, and in the second-pass the oscillation amplitude of the beam 2 was set to a smaller value, ~ 10 nm. The improvements of the magnetic images shown in the **Figure 2(d), (e)** as compared to **Figure 2(b), (c)** are evident. The magnetic images collected in the DT-MFM mode miss any perturbations. The difference of the two modes is crucial and is coming from the tapping part of the scan (in the MFM pass are the conditions in both methods equivalent).

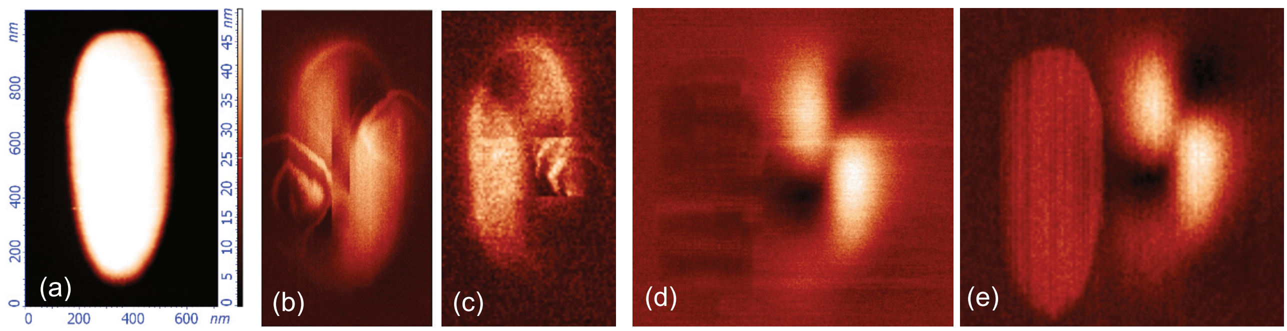

Figure 2 (a) Py ellipse with dimensions 400 nm x 800 nm, thickness 40 nm. Typical magnetic images of the ellipse using standard two-pass MFM method and MESP tip in horizontal (b) and vertical (c) directions, at lift distances 30 nm (b) and 50 nm (c). The images show typical tip-induced perturbations. MFM achieved by the dual-tip method in horizontal (d) and vertical (e) directions at the lift distance 50 nm. Right parts show typical phase shift in elliptical pattern imaged by the magnetic tip, left parts in both figures show the residual of the van der Waals forces depicted by the topography tip.

3. DUAL-TIP FORCE SPECTROSCOPY

3.1. Principle and fabrication of DT-FS

AFM is widely used in biology, in particular, to study cells [14]. It provides true 3D surface topographical information as well as recording of complete force distance curves (FDC). From FDC we can obtain various biophysical properties of materials, such as elasticity, adhesion, hardness, friction, etc [15]. During cell measurement one can meet with the major problem caused by the fact that cells are soft and a possible AFM tip penetration could be quite large. The tip penetration is typically much larger than the radius of the tip apex. The next problem connected with sharp AFM tips is the relatively small contact area. Due to the inhomogeneous cell surface measured rigidity could vary in a wide range. One should make a large number of force measurements to obtain sufficient reliable data. Solution for the above mentioned problems lies in using well defined spherical tips. The larger area of probe-cell contact results in averaging local variation in rigidity compared to the one, measured with a regular sharp probe. Therefore, measurement time become shorter. The well-defined tip geometry is important for regular evaluation of FDC. Such spherical probe can be made by gluing a colloidal spherical particle to the AFM cantilever. Spherical microbeads are made from glass, polystyrene, or PMMA with typical diameter of 1 - 5 micrometres. Preparation of spherical tips is extremely difficult, time consuming and micromanipulator is needed for this purpose. Because of the small bead size there is also a risk that glue can flow over and contaminate it. This may result in an irregular bead shape and thus undefined surface geometry. There have only been published few works related to the topic of preparing blunt spherically-shaped tips. However, a disadvantage of the larger tip radius is loss of image resolution. Here we propose novel method which uses dual-probe to fulfil above mentioned issues. In the first step surface topography is taken with the sharp tip. Then points of interest can be defined in the scan image. Subsequently, the tip with larger radius is used for performing FD curves.

The dual-probe was fabricated by FIB similar to the above-mentioned DT-MFM probe. We modify commercial tip NSC35 from Mikromasch co. The cantilever and tip was cut asymmetrically, so after splitting by FIB, one tip was small and other a bit larger (**Figure 3(a)**). Next, the larger tip was milled from the side to create a plateau at its top (**Figure 3(b)**). The different milling pattern was used for sharpening and blunting process. The small tip was sharpened by simple annular-shaped patterns around the tip apex, etching only a small area of the tip. To get a blunted spherically-shaped tip we had to find proper etching pattern. **Figure 3(c)** shows optimized grayscale pattern where colour of pixels is gradually changed from outer area to the center. White colour corresponds to highest etching speed while black colour represents no etching.

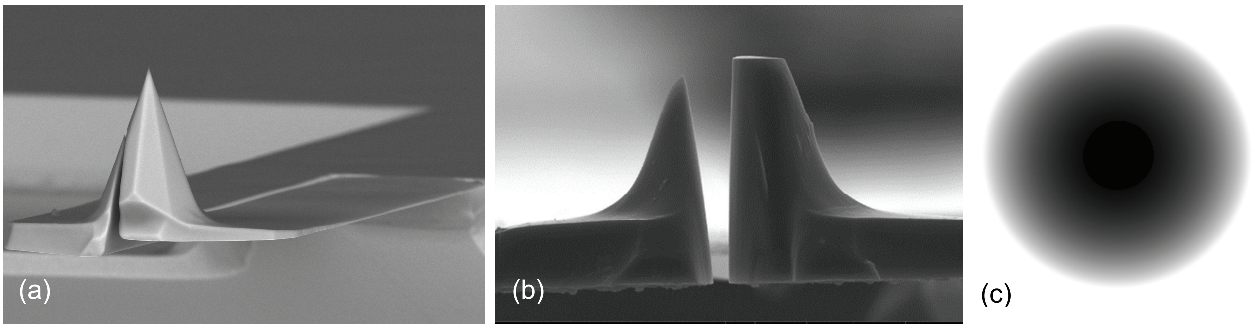

Figure 3 (a) Commercial AFM probe after splitting by FIB, (b) tip apex has to be cut prior to forming into spherical shape, (c) mask pattern used for to form sphere at the large tip

While the sharp tip is being measured, the blunt tip must be displaced away to not touch the sample surface. Switching between cantilevers is realized by means of integrated thermal actuator. Aluminium meander defined on cantilever with blunt tip provides bimorph effect needed for cantilever bending. The meander was defined by electron beam lithography, Al evaporation and lift-off process. To realize EBL direct on cantilever we used our special technique for deposition PMMA rezist on cantilever [16].

3.2. Results and discussion

Figure 4 shows two examples of final dual-tip probes after sharpening and blunting process. We have found that ion beam current of 100pA at acceleration voltage of 30kV gives optimal compromise between etching time and precision of shaping process. Sharpening process allows for preparation of tips with a sub-100 nm curvature diameter. In addition, it is possible to achieve a better aspect ratio than the original tip had before sharpening. Blunting process allows to set the tip curvature diameter in a range of 100 nm - 5000 nm. It is worth mentioning that a commercial tip with eight sidewalls (tip having a octagonal base) is easier to form into spherical shape than pyramidal four-sided (deltoid-shape base) tips. As can been seen from **Figure 4** the end of blunted tip was not shaped into an ideal hemisphere. However, this is not needed for FDC measurements. What is important for FDC is that the tip apex is nicely spherically rounded with a flat surface. The second requirement is that the depth of penetration into cells is less than the tip radius. Thus Hertz model can be apply for FDC evaluation (calculation of Young's moduli of cells or stiffness across the cell). Our blunted tip meets both above mentioned requirements for penetration depth up to 300 nm. The calibration grating TGG1 was used for tip curvature characterization of the blunted tip. As the blunted tip was scanned across the sharp edges of the grating, thanks to the convolution effect, we can see real shape of the tip in tested AFM image. **Figure 5(a)** shows AFM profile of one line taken from the tested image. When the calibration circle is inserted into profile, it is evidence that end of tip represents smooth spherical surface.

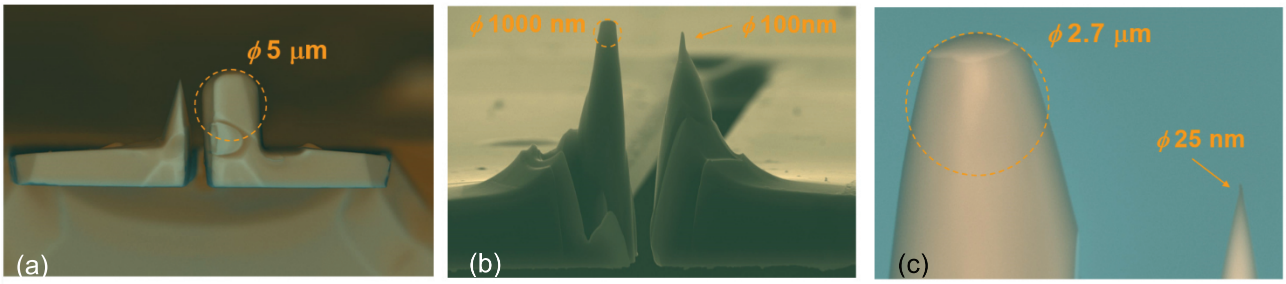

Figure 4 Examples of final probes. Sharp tips with a sub-100 nm curvature diameter can be prepared by a sharpening process. Blunting process allows setting the tip curvature diameter in a range of 100 nm - 5000 nm. (Images were colorized in post-processing).

It is important to note, that for measuring FDC of the cells, the cantilever should have rather low spring constant. This requirement we can easily adjust by lowering cantilever thickness. For example, when the thickness is reduced by half, the constant will decrease by one order. **Figure 3(a)** documents such modification of cantilever thickness where FIB milling was applied from the back side of the cantilever equipped with large tip. This step can be performed prior to tip shaping process or after.

As it can be seen from **Figure 4** the spherical tip is higher than sharp tip, so it is needed to bend it away when measuring topography with sharp tip. Switching to the sharp cantilever can be realized by bimorph effect. **Figure 5(b)** and **(c)** shows our first test to verification of functionality of cantilever bending. The thickness of aluminium meander was 100 nm. Aluminium has been chosen because of its high thermal expansion coefficient mismatch with silicon which yields an efficient bimorph effect. AFM chip with meander was contacted with thin wires and inserted into a SEM chamber. The chip was placed in such a position that we could observe the cantilever deflection from the side. We have increased the electrical power to the chip and measured the actual deflection during the test. **Figure 5(c)** shows almost linear dependence of deflection on heating power up to 10 mW. Achieved deflection of 3 μm is sufficient for our purposes. In the next step we are planning to evaluate heat dissipation from heated cantilever to the neighbouring one. It may be undesirable to have warm sharp tip when scanning topography. If the simulation shows that temperature at the sharp tip is too high we propose to use magnetostrictive effect for the cantilever bending instead of thermal bimorph effect. It was shown that FePd layer placed on cantilever can serve as an active layer and relative small external magnetic field is needed for deflections of two microns [3].

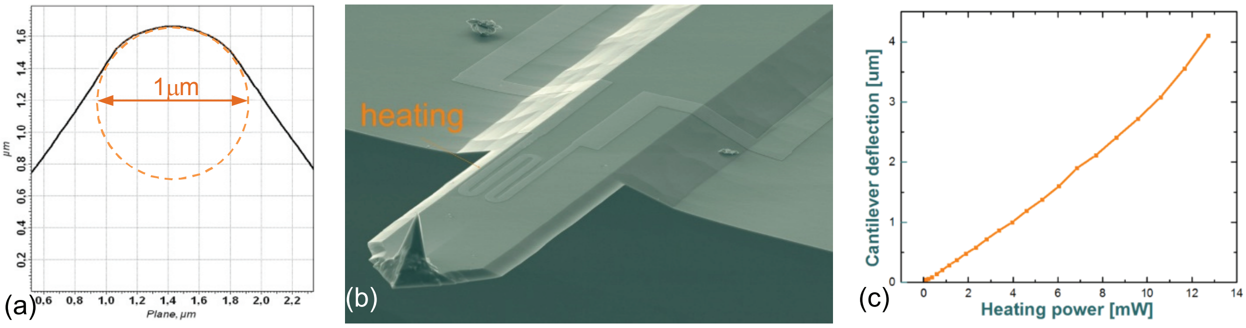

Figure 5 (a) AFM profile of sharp edge of TGG1 calibration grating, which obtained using blunted tip with diameter of 1 μm (see left tip in Figure 4(b)). (b) AFM cantilever with meandered Al heater patterned by direct EBL and lift-off process. (c) Cantilever deflection as a function of electrical power used for the actuation.

4. CONCLUSION

To summarize, we have developed two novel SPM techniques based on dual-tip principle. First technique DT-MFM is aimed for magnetic measurements. Within the DT-MFM method, instead of using one tip for both, topography and magnetic scanning, we use dual-tip with separated functionalities - one is for topography, and the second one for the magnetic scanning. We show that main distortions of the magnetic tip are strongly reduced if the magnetic tip avoids surface touching. We have shown, that contrary to standard MFM scanning, the magnetic image obtained using dual-tip MFM depicts correctly the magnetic state of the permalloy ellipse at the scanning distance of 50 nm. The second method DT-FS was developed for surface topographical information and subsequent quick recording of complete force distance curves. We have shown that by FIB instruments it is possible modify original AFM tip into sharp and blunt tip with spherical tip apex. Switching between cantilevers is realized by bimorph aluminum meander.

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