Focused Ion Beam as tool for atomic force microscope (AFM) probes sculpturing

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Abstract. The fabrication of novel atomic force microscopy (AFM) probes for nanoindentation and nanoimprint lithography (NIL) is presented. Nanomachining induced by focused ion beam (FIB) were employed in order to modify the original tip shape of commercial silicon AFM probes. The FIB-modified probes are used both to perform experiments as to image the corresponding tip-induced surface modifications. With this approach, a relationship between the hardness of a material and the shape of the indenter has been found in the nanoindentation application, and we have obtained information related to the force acting on the mold during its detaching from the polymer film in the AFM-NIL application.

1. Introduction

Scanning probe microscopy (SPM) is universally recognized as a powerful tool for performing a wide variety of experiments with high spatial resolution, because it allows to probe a rich set of different interactions between tip and sample and also to pattern the specimen surface with nanometer-size structures [1-3]. In the recent years, many efforts have been made to fabricate probes with new peculiarity, in particular with the help of FIB technique. Focused ion beams enable reproducible and reliable material processing with high accuracy. Material removal by physical sputtering or chemical-enhanced etching and beam induced material deposition can be used for the processing of structures with dimensions in the micro- to nanometer range [4,5]. In contrast to conventional structuring based on masking techniques, material processing in a direct writing mode by FIB facilitates fast and flexible structuring even on sample with extreme topography, such as SPM probes [6-8].

Nanoindentation and NIL are two techniques that can take advantage from the application of FIB. The first one is used to investigate the surface hardness at the nanoscale [9,10]. The second technique is used to pattern resist film in semiconductor technology to fabricate nanodevices [11,12]. In both applications it is useful performing and subsequently imaging the corresponding surface modification. In this work we describe the fabrication of reshaped silicon AFM probes by means of FIB milling and their applications.

2. Experimental

2.1. FIB nanofabrication

Whole tips modifications were performed with a DualBeam system FEIB235M, combining a Ga⁺ liquid metal ion source (LMIS) FIB and a thermal field emission SEM, working at coincidence on the
sample. Nominal resolutions of the two columns are 7 nm at 30keV for FIB and 2 nm over a wide energy range for SEM. The sample stage can tilt from -10° to 57° with respect the electron beam. All milling steps are performed with ion beam energy of 30keV. Four different apertures were chosen corresponding to nominal beam current values of 10, 30, 100 and 300pA and nominal beam size 10, 13, 20 and 25 nm, respectively. Actual measured currents were 9, 27, 98 and 275pA, respectively.

2.2. Experimental nanoindentation set-up
The experimental apparatus used for nanoindentation is a Digital Instruments EnviroScope Atomic Force Microscope by Veeco® and it allows indenting the sample and imaging it right after the indentation [10]. The set of FIB modified indenters was commercial silicon AFM tips and consequently could not provide a high mechanical profile in terms of hardness and non-deformability. Therefore nanoindentations were performed on a soft substrate, such as Microposit S1813 photoresist by Shipley®. It is a positive photoresist based on a NOVOLAC polymer and its mechanical properties are well known. The photoresist was deposited on a steel substrate by spin-coating and the layer thickness was 1.4 µm.

2.3. Experimental nanoimprint lithography set-up
AFM-NIL experiments were performed employing a SMENA AFM head by NT-MDT® and a home-built Peltier sample heater/cooler, that allows a temperature range from 20° to 120° on the sample itself. Employed polymers were Microposit S1818 photoresist by Shipley®, mr-I 7020 and mr-I PMMA35k by MicroResist® on SiO2 substrate, prepared by spin-coating in order to reach a final thickness in the 200-300 nm range. A pre-patterned by standard NIL polystyrene sample was also employed to exploit the alignment capabilities of our probes. The AFM-NIL process was studied by standard force-distance curves as a function of sample temperature, applied loads and testing different probe geometries.

3. Result and discussion

3.1. Nanoindentation
The pristine geometry of the probe tip is a quadratic pyramid. We have modified it with the aim of transforming the tip into a triangular pyramid, as the nanoindenters usually are. We have proceeded to cut the pristine probe along a plane positioned with suitable different orientations. The angles of the cutting plane are chosen in order to obtain a new tip shape, which can approach the sample perpendicular to its surface during the indentation procedure. In this respect, we always took into account the 12° angle of the AFM probe holder. In figure 1 SEM images of the starting silicon probe (figure 1(a)) and of the three probes obtained by FIB nanofabrication (figure 1(b)-(d)) are reported. In order to know exactly the new geometry of the nanofabricated probes we have used a calibration grid composed of an array of sharp tips (test grating TGT1–NT-MDT®). The measured corner angle were 62° for the first tip, 25° for the second and 97° for the third.

![Figure 1: SEM images of an original silicon AFM probe (a) and FIB modified nanoindenters with corner angle of 62° (b), 25° (c) and 97° (d).](image-url)
The modified probe was used to make five matrices of indentations on the photoresist layer. Each matrix (figure 2(a) and (b)) consists of 16 indentations performed at different loads on a row and repeated column by column. The loads applied on the sample vary from 500 to 2000 nN and the hardness of photoresist layer was obtained from the equation $H = P / Ar$, where $P$ is the maximum load and $Ar$ is the measured residual area.

![Figure 2: Schematic (a) and AFM 3D image (b) of an indentation matrix; (c) Comparison between experimental data (square) and theoretical model (line).](image)

These experimental data are in a good agreement with a shape/size-effect law for nanoindentation, predicted by Pugno [13], as shown in figure 2(c), where this theoretical model is used for fitting experimental results. Details of nanoindentation experiments can be found elsewhere [10].

### 3.2. Nanoimprint lithography

Tips for NIL were obtained starting from commercial silicon non-contact AFM probes, for their high spring constant. In figure 3 SEM images of the modification steps are reported. Positioning the probe at 90° with respect the electron beam, as shown in figure 1(a), the pyramid was flattened (figure 3(a)), in order to have a flat region where create our stamp feature. Then the feature for the stamp and the tip for imaging were created (figure 3(b) and (c)). These modifications were performed considering the angle between cantilever and sample surface during AFM operations. After that, the probe was tilted at 0° with respect the electron beam. In this position the tip was reshaped in order to have good resolution in the AFM images (figure 3(d)) and the stamp was finished. Another probe with a different stamp is shown in figure 3(e).

![Figure 3: sequence of tip modification: (a) flattening of the pyramid; (b) creation of the feature for the stamp; (c) creation of the tip for imaging; (d) reshaping of the tip and stamp; (e) other probe with a different feature for stamp.](image)

With this kind of probes we have performed some preliminary studies on various polymers in order to obtain information about the adhesion properties of a sub-micrometer mold during the pulloff step of a standard NIL process. The sample was heated from room temperature to a value above its glass transition temperature $T_g$. Force distance curves were acquired at these two temperature values (see figure 4(a) and (b)) and at different maximum loads. The “tip-mold” was coated employing the same
process used for anti-sticking treatments of NIL stamps (cleaning and dipping the probe into a silanization solution). During the withdrawal (dashed line) of the tip from the sample surface we observed the characteristic behaviour of adhesion that depends both on van der Waals forces, capillary forces, and possible chemical bonds between the tip and the substrate. Imaging of the indented pattern (figure 4(c)) was done employing the same probe and exploiting its sharp tip at one side of the stamp as imaging tool (see figure 3(d)). This characteristic of our probe was also employed to align the indenting region of the probe on a previous-patterned polymer sample surface.

**Figure 4:** Approach (solid line) and withdrawal (dashed line) force vs. distance curves of the probe shown in figure 3(d) on a mr-I 7020 polymer film (Tg= 60°C) at 24°C (a) and at 115°C (b). (c) AFM image of indentation at different loads on a pre-patterned PS substrate (Tg= 105°C).

### 4. Conclusions

We have illustrated the procedure, based on ion milling by FIB, to create probes with specific tip shape, starting from silicon commercial ones. Modified tips were created for applications in nanoindentation and NIL techniques. In the first case, experiments have demonstrated the theoretical relationship between hardness of the material and shape of the indenter. In the second case, experiments have shown how a NIL process can be monitored in real time employing a FIB modified AFM probe as a mold.

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