SELF-ACTUATED POLYMER-BASED CANTILEVERS WITH SHARP SILICON TIPS FOR HIGH-SPEED ATOMIC FORCE MICROSCOPY

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ABSTRACT

High-speed atomic force microscopy has thus far largely been limited to the use of small cantilevers in liquid, but the technique would greatly profit areas of nanotechnology that require the use of larger probes in air. We present polymer-based cantilevers with a low Q factor, an integrated electrothermal actuator and a sharp silicon tip. The devices can be used directly on commercial AFM setups utilizing a custom cantilever holder. They are suitable for high-speed tapping mode thanks to the high intrinsic damping of their polymer core. The integrated actuator allows for large amplitudes for off-resonance imaging, as well as for clean actuation tunes for tapping-mode imaging.

KEYWORDS

AFM, hybrid MEMS, cantilever, silicon tips, integrated actuator, electrothermal.

INTRODUCTION

The atomic force microscope (AFM) has since its invention [1] become one of the main tools for characterization at the nanoscale. Its main drawback is the limited acquisition speed: obtaining an image can take up to several minutes. The achievable speed is often dictated by the performance of the cantilever. This issue has been approached by using smaller cantilevers, below 10µm in length. In the case of tapping mode, the high resonance frequency (f_0) of such small cantilevers helps to increase the imaging speed, which is frequently limited by the cantilever bandwidth (proportional to f_0/Q [2]). When imaging in liquid, a low Q environment, video rate acquisition has been achieved [3]. In the case of off-resonance tapping modes (ORT, also called Pulsed Force mode [4], PeakForce QNM [5], etc.), the imaging speed is usually limited by the rate at which interaction curves can be obtained (ORT rate). Most systems use the z-scanner for actuation and are thus limited to ORT rates well below the scanner resonance, typically a few kHz. Directly actuating the cantilevers allows to circumvent this problem and increase the rates. For instance, small cantilevers with their low thermal mass can be driven efficiently with a pulsed laser and increase ORT rates to hundreds of kHz [6].

Most AFM systems however still use relatively large cantilevers. Our group previously proposed polymer cantilevers to increase the tapping mode speeds in air [7]. The polymer introduces a lot of material damping, drastically reducing the Q factor. However, the drawback are complex tuning curves, the so-called “forests of peaks”. Here, we propose a new cantilever that exhibits clear cantilever tunes for tapping mode and can also be actuated off resonance for fast ORT. We use a trilayer technology, where the polymer is sandwiched between two hard films and placed on a silicon chip, enabling a much clearer inertial tune [8]. In addition, we integrated an electrothermal heater to actuate the cantilever directly, further improving the tune as well as enabling high-speed ORT. Finally, we use a previously established fabrication method [9] to create sharp silicon tips on the cantilevers.

FABRICATION

The cantilever fabrication (Figure 1 – a) is based on the hybrid MEMS process [10], which uses adhesive wafer bonding to form a trilayer structure consisting of a thick polymer core between two hard thin films. Electronics such as actuators or sensors can be freely integrated and will be insulated from the environment by the thin films.

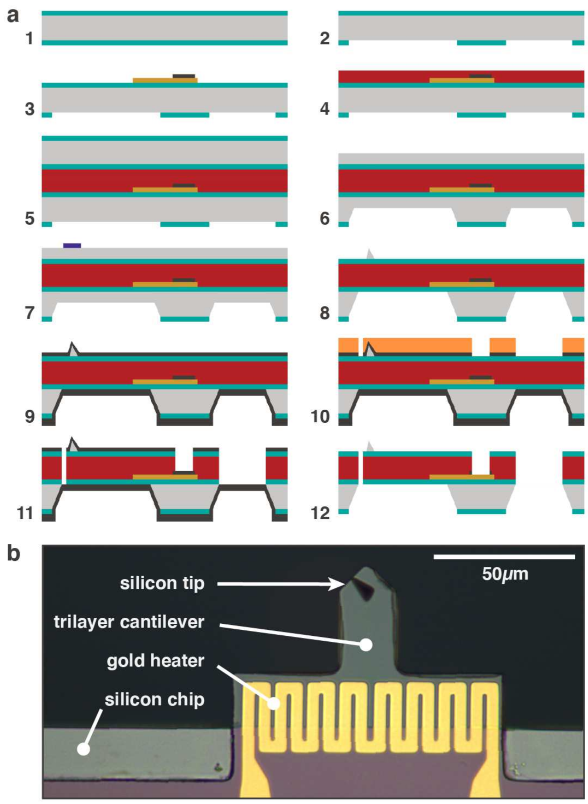

Figure 1: Device fabrication. a – The fabrication process consists of 5 masks: backside chip (2), gold trace and aluminum pads (3), tips (7) and cantilevers (10). Materials: Si (grey), SiN (turquoise), Au (gold), Al (black), BCB (red), SiO_2 (purple), PR (orange). b – The result is a trilayer cantilever made of a thick polymer core sandwiched between two silicon nitride films. It includes a heater for actuation (gold meander) and a sharp silicon tip. The design footprint can be chosen freely. Here we opted for a wide actuator and a 40x20µm cantilever.

Integrated Electrothermal Actuator

The first part of the process focuses on the integrated electrothermal actuator. They are fabricated on top of the hard thin film (low stress silicon nitride, SiN). The starting materials are two double-side polished, <100> oriented, 380µm thick silicon wafers, upon which 20nm of SiN are deposited through LPCVD (step 1). Photolithography is used to pattern alignment marks and a chip release design onto the backside of one of the two wafers. The design is dry etched through the SiN, before the photoresist (PR) is stripped in oxygen plasma (step 2). Next, two subsequent lift-off steps are used to fabricate the heater on the top side of the wafer (step 3): the first creates the metal meander out of 190nm thick gold on a 10nm chromium adhesion layer, while the second forms a 100nm thick aluminum pad that will act as an etch stop later in the process. The metals are deposited through evaporation.

Glue Bonding

Next, the trilayer structure is formed, encapsulating the electronics. This is achieved with Benzocyclobutene (BCB), a viscoelastic material available as CYCLOTENE 3022™ from DuPont™. This polymer can be spin-coated at thicknesses ranging from 1 to 26µm, used to bond wafers, as well as dry etched using CHF_3 chemistry; all of these properties are crucial to our process. First, both wafers are coated with 1µm of BCB (step 4), then they are bonded together (step 5).

Tip and Chip Fabrication

We make use of both of the wafers in the next steps: the first to form chips that will allow us to handle the cantilevers, the second to create the tips. To do so, the SiN on the top side is first stripped through dry etching. Then, the silicon is etched in KOH until the top wafer is thinned to 10-20µm (step 6). The chips on the bottom wafer are forming at the same time. Next, a pentagonal mask is created [9], it will be under-etched to form the tips. To do so, a layer of SiO_2 is evaporated, then patterned through photolithography and wet etching in BHF (step 7). Finally, the wafers are put back into the KOH solution for the chips and the tips to form. The resulting trilayer membrane (step 8) has to be patterned in order to form the cantilevers.

Cantilever Fabrication

The membrane being too fragile to perform the remaining photolithography and dry etching on, a thick aluminum layer is evaporated onto the backside for added stability. In addition, 300nm of aluminum is evaporated on the frontside to create a hard mask for the subsequent dry etch (step 9). The following lithography is done with a thick resist (20µm) to ensure tip coverage. The wafer is exposed to a short, low-power oxygen plasma for hydrophilicity before the aluminum is wet etched (step 10). The resist is wet stripped, then the trilayer membrane is etched to form the devices. The etch lands on the backside aluminum layer as well as on the aluminum pad above the gold track (step 11). The dimensions and shape of the finished cantilevers can be chosen freely (Figure 1 – b). Finally, the remaining aluminum is wet stripped, releasing the cantilevers and opening an access to the gold pad that will be used to wire-bond the heaters (step 12).

RESULTS

Tip Characterization

To assess the sharpness of the tips, we first imaged the tip profile with a scanning electron microscope (SEM, Figure 2 – a). We then estimated the tip shape by imaging a TipCheck sample (polycrystalline titanium roughness sample, Figure 2 – b) using a Bruker AFM (MultiMode V, Nanoscope V controller, E-scanner). The tip sharpness can be extracted from the AFM images using the blind tip estimation algorithm [11] of the Gwyddion software [12]; the entire procedure is described elsewhere [8]. We took tapping mode images with 15 different cantilevers and the following imaging parameters: 2×2µm scan size, 1024x1024 pixels and 2Hz scan rate. The tips exhibited a radius of 17±2nm at 10nm below the apex.

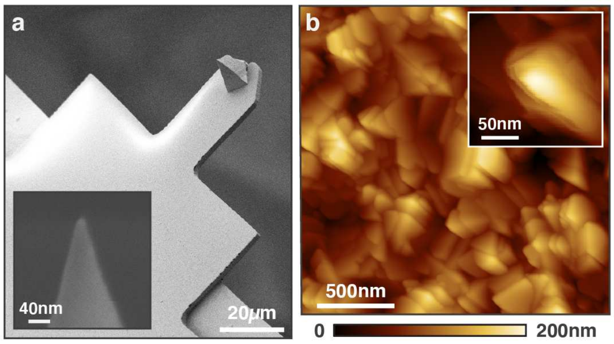

Figure 2: Tip evaluation. a – SEM micrograph of a fabricated cantilever with the integrated silicon tip. The inset shows a side-view of the tip apex, indicating a tip-radius below 20nm. b – AFM image of a titanium polycrystalline roughness sample (Bruker. The inset shows the tip shape, obtained with the Gwyddion blind tip estimation algorithm. The radius at 10nm from the apex is estimated at 17nm.

Tapping Mode

In amplitude modulation tapping mode, the cantilevers are actuated at a fixed frequency close to their resonance. When approaching the surface, the resonance peak shifts slightly to higher frequencies, resulting in a decrease in amplitude at the drive frequency. The change in amplitude is detected with a lock-in amplifier and used as feedback setpoint during imaging. Having a clean actuation of the cantilever resonance is essential for high quality, low force AFM imaging. This is however not always the case, and an unclean resonance tune can result in imaging instabilities. Spurious resonance can occur in the driven frequency spectrum if resonances of the mechanical drive or of the cantilever holder are superimposed on the inherent resonance spectrum of the cantilever. This effect is very prominent with cantilevers having a low Q-factor like polymer cantilevers, or when cantilevers are driven in fluid where the environmental damping dominates over the internal damping in the cantilever. A number of direct cantilever drive mechanisms have been developed for AFM imaging in fluid such as photothermal [13], Lorenz-force [14], magnetic [15] or electrothermal actuation [16]. The tri-layer technology provides an easy way to add an electrothermal actuator. The frequency tune clearly improves when actuating the cantilever directly than when doing so with the external tapping piezo (Figure 3 – a).

We used the cantilevers to image a porous membrane used in battery separators (Figure 3 – c, d, e). The small, suspended fibers are easily displaced by the AFM tip, requiring a well-tuned AFM feedback loop and a sharp tip to be resolved properly. We used a Bruker AFM (MultiMode V, Nanoscope V controller, E-scanner, and custom cantilever holder, Figure 3 – b) with a 40x20µm trilayer cantilever (f_0 = 543kHz, Q = 92) to obtain an image of 2×2µm scan size, 1024x512 pixels, at a 1Hz scan rate.

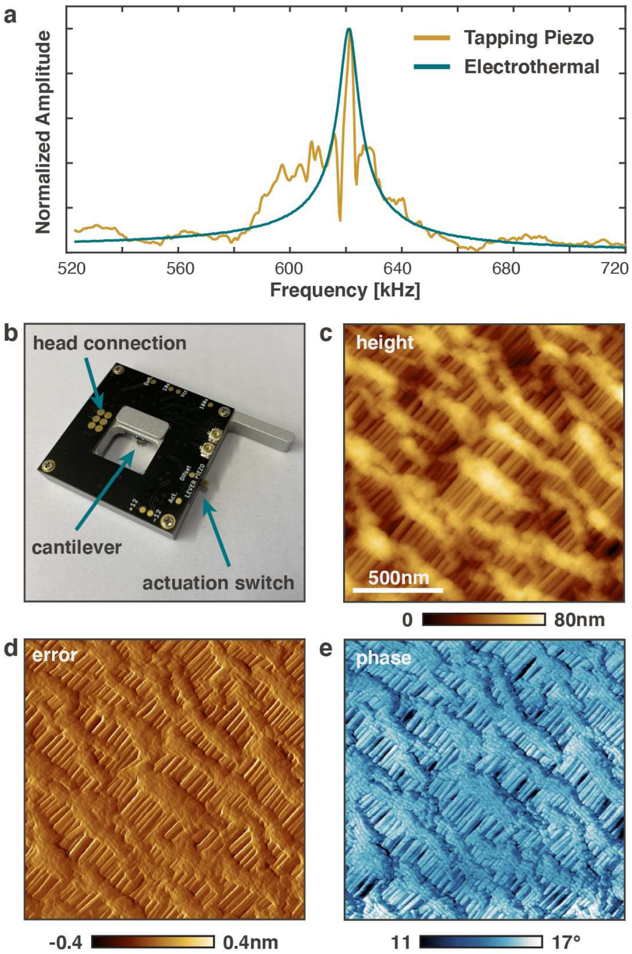

Figure 3: Tapping mode in air with electrothermally actuated tri-layer cantilevers. a – A cantilever tune obtained with a tapping piezo exhibits multiple peaks as a result of mechanical couplings. The tune obtained with the integrated electrothermal actuator shows a clean frequency response. b – Custom cantilever holder for Bruker Multimode systems. c, d, e – AFM image of Celgard® shows both the sharpness of the silicon tip and the good tapping mode imaging performance.

Off-Resonance Tapping Mode

ORT modes have become popular in recent years due to their ease of operation and their ability to extract the mechanical properties of the sample. While ORT modes are much faster than the previously used force volume method, they are still limited the ORT rate. Most AFM systems use the z-piezo of the scanner to create the sinusoidal motion between the tip and the sample. This limits the rate to a few kHz, below the first scanner resonance. Here, we use the integrated electrothermal actuator of our cantilevers to increase the ORT rate to 10kHz. At this frequency, the probe can achieve amplitudes above 100 nanometers and apply the relatively large forces required to obtain a good material contrast on stiffer samples.

We used a tip-scanning AFM (Digital Instruments Dimension5000, home-built AFM controller and custom cantilever holder, Figure 4 – a) with a 60x20µm trilayer cantilever (f_0 = 271kHz, k = 6.4N/m) and a force setpoint of 200nN to obtain an image of 10×10µm scan size, 1024x512 pixels at a 10kHz ORT rate and 0.5Hz scan rate. The imaged sample is a fiber-reinforced polymer [17] based on glass fibers surrounded by epoxy. The mechanical properties channels reveal a good contrast between the hard fibers and the softer epoxy (Figure 4 – b, c, d).

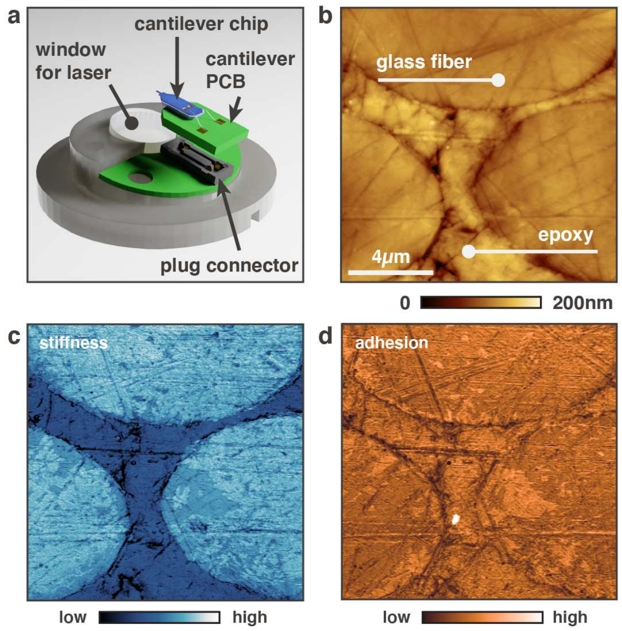

Figure 4: ORT in air with electrothermally actuated tri-layer cantilevers. a – Custom cantilever holder for the Dimension5000 tip scanner. The cantilever is glued and wire-bonded onto a small PCB that can be plugged onto the holder. b, c, d – Image of a polished surface of a fiber-reinforced polymer, showing topography, stiffness and adhesion of the surface. The material property channels clearly distinguish the hard glass fibers from the softer epoxy. They also reveal small patches of a softer material on the glass fibers, likely a polishing artefact.

DISCUSSION

Fabrication

We decided to use the trilayer process for its versatility. For instance, the integration of electronics or tips is entirely decoupled from the device shape and dimensions. This allowed us to design a cantilever with a wide base actuator incorporating a metal heater, while at the same time including a silicon tip whose fabrication is based on an entirely different process.

The tip fabrication is achieved by under-etching a pentagonal hard SiO_2 mask. The five sides of this mask need to be oriented according to the crystal structure of the silicon wafer. Because of this, the tips seen on the cantilever (Figures 1, 2) are at an angle with the cantilever. A possible solution is to bond the top wafer at a 45° angle, so that the final tips can be aligned with the cantilevers.

To obtain a good tip sharpness, we used a sharpening method. Since these tips have three sides, they are self-sharpening when etched in KOH. Thus, we dipped the finished wafer with the devices into HF (1%) to remove the native oxide on the silicon tips, then into KOH for a short time. This yields reliably sharp tips and could in theory be used to resharpen used cantilevers. It should be noted that since the devices contain a polymer, oxidation sharpening cannot be performed because of the high temperatures involved in this step.

Imaging

While cantilevers containing polymers perform faster in tapping mode thanks to the added material damping, they often exhibit a forest of peaks when being tuned acoustically with the tapping piezo. Here, we show an improved tune of the cantilever in air, revealing one advantage of the integrated actuator. This improvement would be much more marked for cantilevers used in liquid, where driving the cantilevers acoustically is more difficult. The proposed cantilevers can be used in conductive liquids as the electronics are encapsulated by the SiN films.

Another advantage of the integrated actuator is that large amplitudes can be obtained at frequencies well below the cantilever resonance. This is crucial to improve the speed of ORT imaging: by actuating the cantilever directly, we are no longer limited by the resonance of the scanner. In the 10kHz ORT measurements (Figure 4), we were limited in scanning speed by the other components of the AFM, such as the scanner and the feedback loop. The challenges to achieving even higher ORT rates are two-fold. On one hand, the cantilever ringing (after snapping of the sample surface) increases with high ORT rates and needs to be limited. The intrinsic damping of the polymer core in our cantilevers enables fast energy dissipation and will help us for that. On the other hand, the actuation efficiency of the thermal heater decreases with higher frequencies. Here, we may start integrating piezoelectric actuators into our devices, as they exhibit a constant response over a very large frequency band. The versatility of the trilayer fabrication process gives us the ideal platform to tackle these issues.

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