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Research Report

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Abstract

Thermomechanical writing occurs as Joule-heated, cantilevered tips imprint nanometer-scale indentations (bits) in a 50-nm-thick polymer (PMMA) film. Thermal data reading incorporates the same cantilevers operated in a mode to detect a temperature change when a tip follows the contour of a previously written bit. Binnig et al. [1] demonstrated single-cantilever writing and reading density at 400 Gbit/in^2. A micromachined 32×32 cantilever array has been fabricated [2] and has demonstrated parallel read/write operation at 150 Gbit/in^2 [3]. Although much progress has been made to develop a thermomechanical data storage device [4], the fundamental process of thermomechanical bit formation is not well understood. Furthermore, macroscopic polymer rheological parameters are unlikely to apply as the bit size approaches the polymer molecule radius of gyration. We have performed detailed investigations of the thermomechanical storage processes by applying atomic force microscope (AFM)-based force detection during thermal operation. We examine the thermomechanics of polymer indentation with respect to time and temperature of interaction. This work impacts the operation of AFM cantilevers for combined thermal writing and reading and understanding of fundamental polymer mesoscopic transport.

INTRODUCTION

The Millipede is a high-density, massively parallel AFM-based storage project based on a highly integrated microelectrical mechanical system (MEMS). A 32×32 array of AFM-style cantilevers perform simultaneous read, write, and erase storage operations on a supported thin film of polymer. Each lever is individually addressed and controlled in a multiplexed control scheme that creates and senses bits with a thermomechanical interaction with the polymer. Data bits are written when a Joule-heated cantilever is forced into the polymer film, leaving a permanent impression. Reading of these indentations is performed by contact-scanning a moderately heated cantilever over the bit and sensing a change in temperature when the tip traces the contour of the bit. By the nature of its operation, thermomechanical storage gives rise to various tribological and rheological issues of a sharp tip interacting with the thin polymer film. In this paper we address the issue of adhesion due to transfer of material to the tip for various operating conditions of the device.

Several important qualities of the polymer storage medium allow it to perform its function as a mechanical receptor of information. First, it retains its amorphous form down to feature sizes smaller than its nominal bulk radius of gyration as demonstrated by bit features (such as a pileup ring around an indentation) of 40 nm or less [1], and thus no natural length scale has been observed that would place an upper limit on the storage density. Second, the mechanical state of the polymer changes sharply as a function of temperature at a relatively low temperature above the ambient. This means that the power required to create bits, that is, to displace the medium surface and create the representation of information, is minimized. The exponential nature of the change in mechanical strength through the glass transition is reminiscent of a switching action. The *on* state of the switch is realized by thermal-assisted indentation stressing, followed by a rapid quenching of the polymer, whereas the *off* state, or bit erasing, is achieved by thermal relaxing of that stress by, say, bringing a warm tip near the bit for sufficient time.

It is well known that the linear viscoelastic mechanical response of bulk PMMA is governed by both the time and temperature of the interaction [5, 6]. Successive states of glass, glass transition, rubber, and viscous flow can be probed by increasing temperature or extending the time of interaction [7]. Each state corresponds to a particular form of molecular motion of increasing activation energy and longer length scale. For thermomechanical data writing, we wish to have a compliant material that does not involve transferring material to the indenting tip. This corresponds to the polymer rubbery state with low mechanical strength but no viscous flow. Data reading should occur with the polymer in a mechanically stable glassy state to avoid wear of the material. Although conditions of time and temperature are known for these states in the bulk, the polymer behavior in a confined thin film is likely to be different due to different restrictions on the molecular motion. In the following we show the existence and give the operational parameters for such interactions at this length scale.

EXPERIMENTAL DETAILS

To simulate the operation of various possible Millipede cantilever forms, a large variety of single-cantilever chips were designed and microfabricated in house. The cantilever used in this work had the form and dimensions as indicated in Figure 1, similar to that of the existing Millipede chip [2] cantilevers in all but two aspects: the thickness was much less at ∼200 nm and the integrated tip was a short 200 nm "in-plane" pyramid at the very forward nose point of the lever. The cantilever had a calculated spring constant of 0.01 N/m and a

measured resonance frequency of about 59 kHz in air. Heavily boron-doped (10^{20} cm^{-1}) legs provided a conducting path to a lightly doped (10^{17} cm^{-1}) nonlinear resistive heater region near the tip. The current and resistance versus DC voltage characteristics of the heater in air are shown in the plot in Figure 2. The resistance curve initially increases with voltage due to increased phonon scattering at higher temperatures, but then peaks and decreases again as the thermally liberated charge carrier density increases. A calibration of heater resistance versus temperature was done in an oven, where it was found that the peak resistance occurred at 550 °C. All electrical measurements on the heater were performed by recording voltages in the bridge circuit as shown in Figure 2.

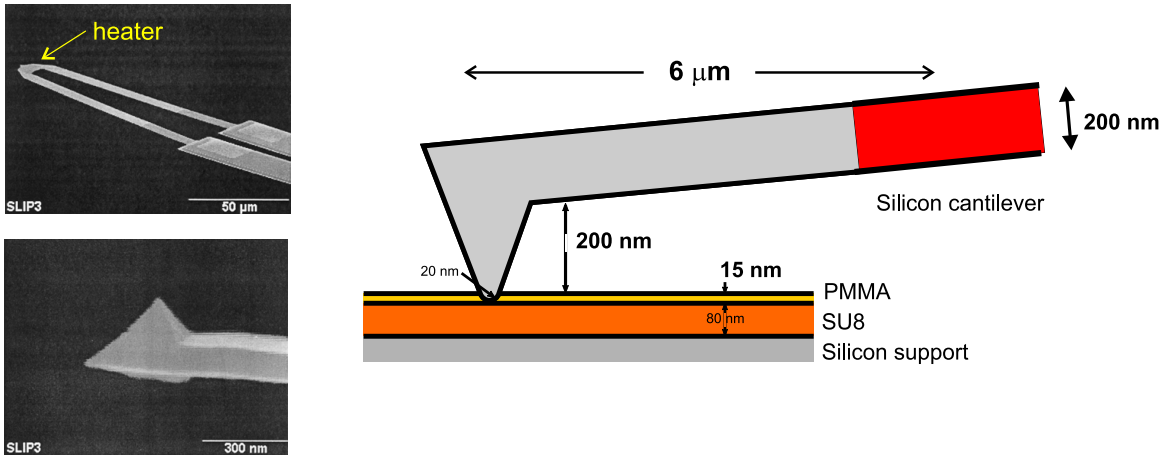

Figure 1: Left: Scanning electron micrograph of cantilever extending from support structure and detail of the integrated trigonal tip. Right: Schematic of the end of the cantilever in contact with the layered storage medium.

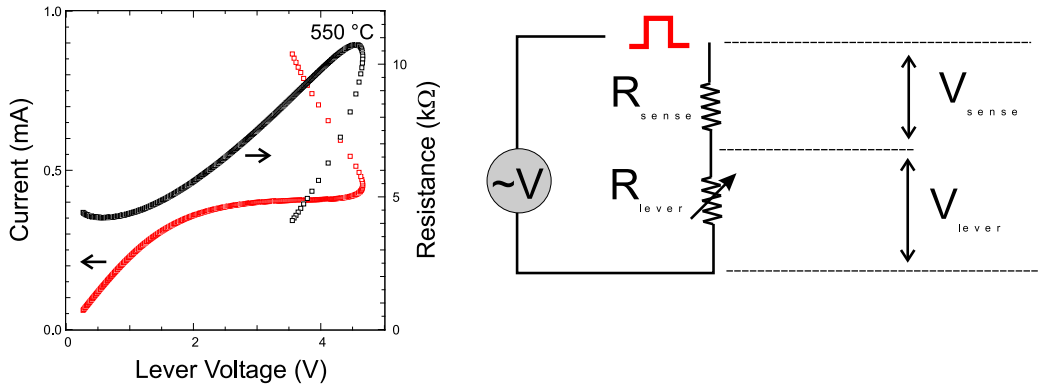

Figure 2: Left: The nonlinear current and resistance versus voltage behaviour of the cantilever heater. The heater temperature at its peak resistance was 550 °C. Right: The bridge circuit from which all measurements were made. The sense resistor had a value of 5.1 kΩ.

The storage medium sample consisted of a 15-nm layer of poly(methal methacrylate) (PMMA) spun-cast on top of a 80-nm layer of highly crosslinked SU8 epoxy supported on a silicon wafer substrate with native oxide covering as shown on the right-hand diagram in Figure 1. The lithographic-grade PMMA had a weight average of $M_n \approx 650,000$ g/mol with a broad chain weight distribution and a corresponding mean radius of gyration of about 60

nm in the bulk melt. The glass transition for a bulk sample was determined by a differential scanning calorimeter (DSC) measurement to occur at around 115 °C.

A probe microscope test stand was built with a fixed cantilever holder and sample scanned in three dimensions. Thermal drift in the system was low enough to allow operation without feedback regulation for several hours. An optical beam deflection system allowed detection of forces on the cantilever during thermomechanical operations of bit reading and writing. All experiments were conducted with well-defined and reproducible loading conditions of the cantilever on the surface by performing force versus distance style approach curves before and after a reading or writing operation. Material transfer to the tip was detected as the magnitude of hysteresis between the approach and retraction portion of such curves.

DISCUSSION

The left-hand plot in Figure 3 is a typical approach curve for the cantilever heated at a typical DC readback power of 0.25 mW. The piezoelectric scanner stage displaced the polymer surface approximately 5 μm towards the lever and then back again within a time of 350 ms, with an overall contact time with the polymer of about 100 ms. In the upper curve ("Thermal") the resistance of the heater pad drops 50 Ω as the cantilever approaches the sample and is cooled by conduction of heat through the air into the polymer. At -2 μm the tip continuously touches the surface, and a relatively minor change in the resistance occurs over the remaining approach distance as the lever pivots about the tip and the separation of the heater and surface reduces much more slowly. Upon retraction, the resistance trace closely follows the approach curve except for a small hysteresis and jump-from-contact phenomenon. The beam deflection signal ("Optical") tells a complimentary story. The point of contact and pivot of the tip on the surface at -2 μm is registered as a sudden increase of the deflection signal. During retraction the deflection mimics the corresponding resistance signal, perfectly exhibiting the same 500 nm (or about 5 nN) hysteresis and jump-from-contact effect. The 630-nm oscillations of the optical signal in the region that the tip is not in contact with the sample are a superimposed interference artifact of the imaging system. The extreme thinness of the reflecting cantilever permits some light transmission that reflects from the sample surface and back into the imaging optics. There is an initial lagging mismatch between the approach and retraction deflection curves, which is also present in the resistance signal. The presence of this mismatch in the non-contact region means that most of what appears as contact hysteresis is instead a scanner hysteresis artifact, which disappears by the time maximum retraction is reached. This is confirmed by approach curves that do not touch the surface but exhibit a similar behavior. We note in passing that the relative resistance $\Delta R/R$ per nm thermal sensitivity of about 1.4×10^{-5} (indicated by the dashed tangent of the resistance curve at the point of contact) is about one order of magnitude larger than a typical piezoresistive cantilever sensitivity.

Characteristic changes of the contact hysteresis will occur when polymer material is transferred to the tip. The right-hand plot in Figure 3 shows an example of such an event. The approach curve was recorded after 6 h of continuous scanning over the polymer surface with the lever operated in thermal read mode and a lever voltage of 1.5 V. Note the large and complex pulloff hysteresis in comparison to the left-hand panel. The experiment shows that at bit-reading temperatures, transfer of polymer material is a slow but non-negligible process. Keeping the tip at room temperature, on the other hand, no enhancement of the contact hysteresis was observed after extensive scanning. Hence, we conclude that transfer of polymer material is quenched at sufficiently low tip temperatures.

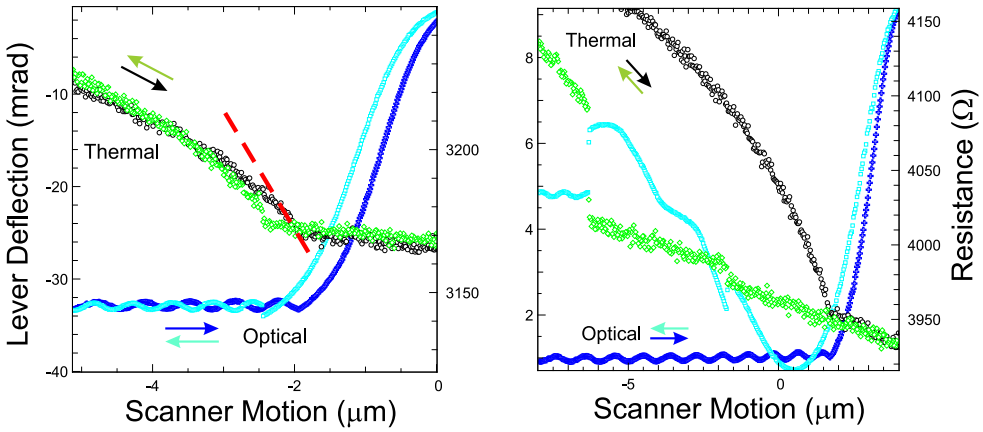

Figure 3: Left: Resistance and optical deflection versus scanner motion for 350 ms approach and retraction cycle of 0.25 mW heated cantilever to polymer sample. Right: Approach curve for cantilever after 6 h of thermal read-scanning of sample.

Finally, we investigate whether a similar transfer phenomenon occurs at the high temperatures and forces, but short periods of time, associated with bit writing. To do this we performed five identical approach curve experiments at different locations on the polymer surface. At the point of maximum loading in each we applied successively higher power writing pulses of 10 μs in duration. The region over which the five bit-writing events occurred was imaged at the same loading conditions as the bits were written, the results of which can be seen in the thermal (resistance) and optical deflection images in the lower half of Figure 4.

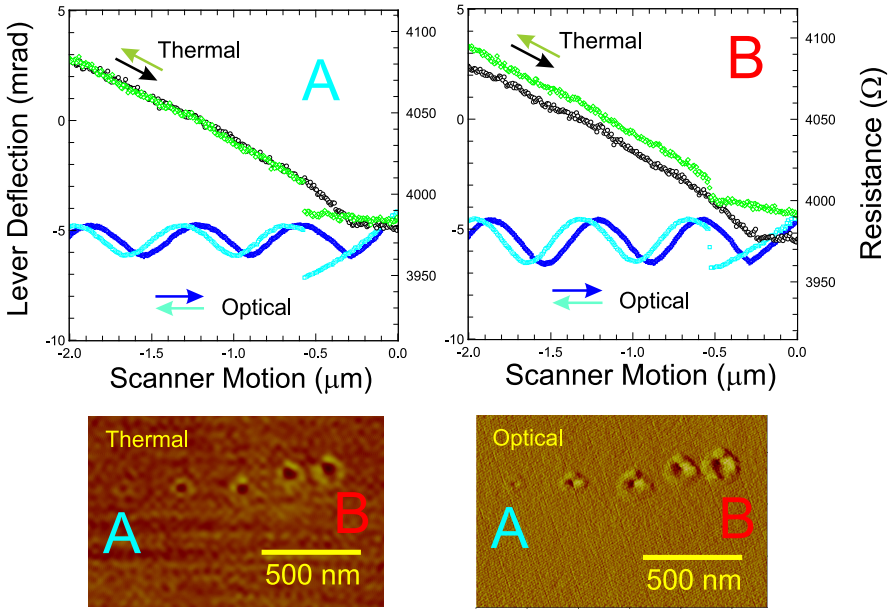

Figure 4: Lower: Thermal (left) and optical deflection (right) images of five nanoindentations (bits) written with 10 μs pulses of increasing power from left to right in 15 nm of PMMA on an 80-nm SU8 buffer layer. The smallest indentation (A) has a pileup diameter of 130 nm, whereas the largest (B) is about 220 nm. Upper: The resistance and optical deflection approach curves corresponding to the writing of bit (A) (left) and (B) (right).

The consumed pulse energy ranged from 20 to 35 nJ, which produced peak final heater temperatures from 500 to 700 °C, as determined from the independent resistance-versus-temperature calibration. The $\Delta R/R$ per nm thermal sensitivity for these experiments was $3.1 \pm 0.3 \times 10^{-5}$ due to a higher read heating power of 0.5 mW. By this calibration, the depth of the three larger bits in the thermal version of the scanned image was 21 ± 4 nm and therefore these bits were written primarily into the PMMA layer and not into the SU8. The apparent depth of the smallest bit, at around 5 nm, meant this event was just on the verge of writing. The 420 ms approach cycle for the smallest bit (labeled **A**) shows minor hysteresis of 300 nm (or about 3 nN) similar to that of the first approach curve with no bit-writing pulse discussed above. As the bit-writing power was increased, the approach curves evolved continuously into the form shown for bit **B**, but no hysteresis was introduced. Note that the retraction resistance curve is hotter than the approach curve for bit **B**; this is due to residual heat from the extremely high-temperature pulse, which does not dissipate fully within 200 ms. We conclude that the transfer of polymer material cannot be activated on the microsecond time scale even at these elevated temperatures.

In summary, we have shown that high-speed, high-temperature nanoindentation of a thin PMMA film does not induce transfer of polymer material to the indenting tip. Material can be transferred to the tip if sufficient time is given such as raster scanning for a number of hours at bit-reading temperatures.

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