# HEATED ATOMIC FORCE MICROSCOPE CANTILEVERS WITH WEAR-RESISTANT ULTRANANOCRYSTALLINE DIAMOND TIPS

*H. J. Kim[1], N. Moldovan[2], J. R. Felts[1], S. Somnath[1], Z. Dai[1], T. D. B. Jacobs[3],*
*R. W. Carpick[3], J. A. Carlisle[2], and W. P. King[1]*
[1]University of Illinois at Urbana-Champaign, Urbana, USA,
[2]Advanced Diamond Technologies Inc., Romeoville USA,
[3]University of Pennsylvania, Philadelphia, USA

## ABSTRACT

We report a wear-resistant ultrananocrystalline diamond (UNCD) tip integrated onto a heated atomic force microscope (AFM) cantilever. The batch-fabricated UNCD tips have tip radii of about 10 nm and heights up to 7 μm. The tips were wear-resistant throughout 1.2 m of scanning over a silicon grating at a force of 200 nN and a speed of 10 μm/s. Under the same conditions, a silicon tip was completely destroyed. When used for thermal imaging, the UNCD tip heated cantilever has a vertical imaging resolution of 1.9 nm. Finally, we demonstrate thermal nanolithography of hundreds of nanostructures of polyethylene.

## INTRODUCTION

Atomic force microscope (AFM) cantilevers with integrated heaters have been used for data storage [1], nano-imaging [2], and nano-manufacturing [3], and material property measurements [4]. An important requirement for AFM cantilevers is stability of the tip shape, which can be problematic when the tip, usually silicon or silicon nitride, is scanned over a hard surface. The tip stability improved by a tip coating or the use of novel tip materials [5-7]. Here we report wear-resistant UNCD tips integrated onto a heated AFM cantilever and an array of heated AFM cantilevers.

Diamond is an attractive material to be used as a tip, since it has high hardness, chemical stability, low friction coefficient, low work function, and low adhesion [8]. Various methods have been proposed to fabricate diamond AFM cantilever tips. For example, focused ion bean milling can generate sharp diamond tips, although it is expensive and relatively slow [7]. Molding can batch-fabricate monolithic diamond tips [5], but this process may not be well-suited to integrate electronic devices into the tip and cantilever, and often generates wedge shaped defects. Another batch fabrication method is to coat silicon tips with a thin layer of diamond [6], but the typical tip radius (>50 nm) may be larger than is desired, since the coated diamond layer increases the radius of the final tip. Batch fabrication of diamond tips using reactive ion etching (RIE) is a simple process [9] that can provide good control over tip dimensions, such as tip radius and height, thus generating sharper and taller diamond tips than other fabrication methods.

This paper reports the integration of UNCD tips onto a heated AFM cantilever and cantilever arrays using UNCD coating and RIE sharpening. We investigate cantilever electrothermal properties and tip wear. In addition, we demonstrate the use of UNCD tip heated cantilevers for thermal topography imaging and nanolithography.

## DESIGN AND FABRICATION

Figure 1 shows the UNCD tip heated cantilever fabrication steps. The cantilevers are fabricated from doped silicon-on-insulator wafers. The cantilever fabrication starts with a cantilever formation using inductively coupled plasma (ICP) deep RIE followed by two separate phosphorus doping steps at different concentrations. The cantilever free end was low doped (~$10^{17}$ $cm^{-3}$) to act as a resistive heater, while cantilever legs were high doped (~$10^{20}$ $cm^{-3}$).

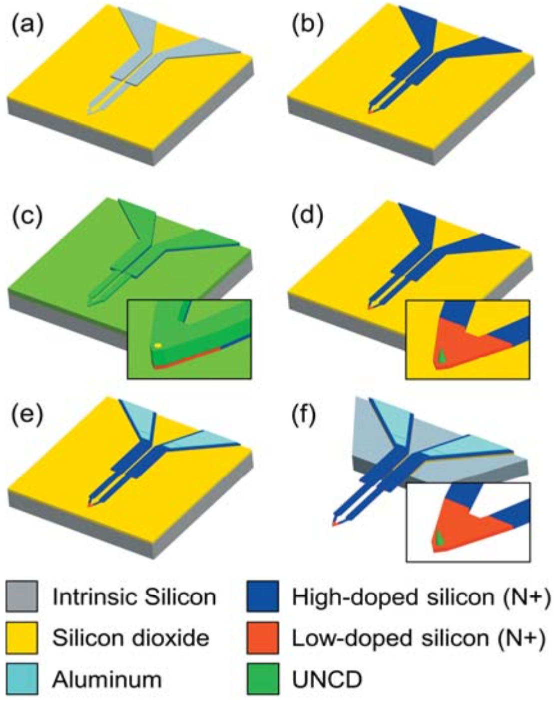

*Figure 1: Summary of fabrication steps. (a) Cantilever formation with ICP-RIE. (b) Low dosage and high dosage phosphorus implantation. (c) UNCD growth via hot filament CVD. (d) UNCD tip etch using RIE. (e) Aluminum contacts formation. (f) Final device release via backside through etch and HF wet etch.*

978-1-4673-5655-8/13/$31.00 ©2013 IEEE

For UNCD tip formation, the silicon wafer substrates were seeded with diamond nanoparticles by ultrasonication and a 7-µm-thick UNCD layer was grown by hot filament chemical vapor deposition (CVD). A protective $SiO_2$ mask was patterned to form 3-µm-diameter precursor caps, and the exposed UNCD was etched with ICP-RIE in $O_2/SF_6$ plasma. The differential etching rate of UNCD to $SiO_2$ of about 15:1 allows the gradual etching of the protective $SiO_2$ mask until it is completely removed, resulting in ultrasharp UNCD tips. Aluminum contacts and a backside etch completed the fabrication. A single 100-mm-diamter wafer could produce about 500 cantilevers with good tip sharpness across the wafer.

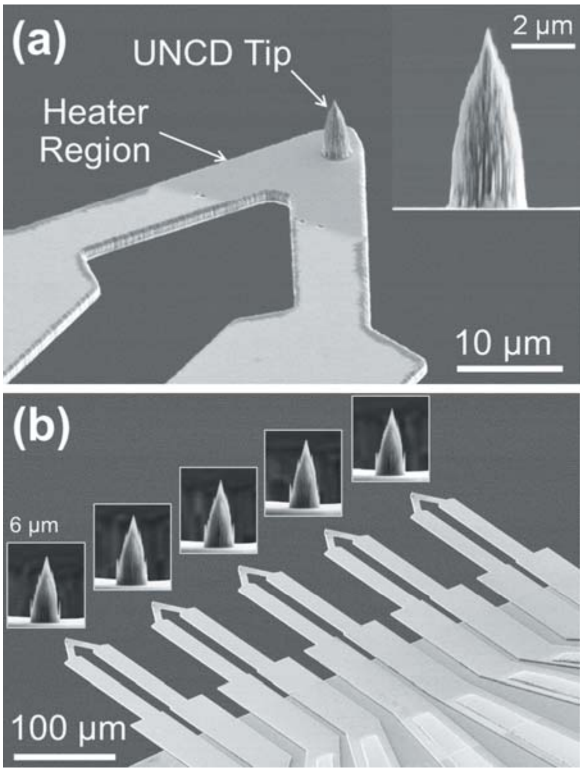

*Figure 2: (a) A heated AFM cantilever and (b) an array of heated AFM cantilevers with UNCD tip integrated onto a low-doped heater region.*

Figure 2 shows scanning electron microscope (SEM) images of a heated AFM cantilever and an array of cantilevers with UNCD tip integrated onto a heater region. Fabricated UNCD tips have a typical apex radius of about 10 nm and an overall aspect ratio of about 4:1. The tip is sharper than diamond-coated tips and taller than most tips fabricated onto heated AFM cantilevers using other techniques [1, 6], thus can image taller topography features. The cantilever array consists of five individually-controllable identical cantilevers, with a pitch of 110 µm. All cantilevers in the array have nearly identical tip profiles.

## THERMAL AND MECHANICAL CHARACTERIZATION

We characterized the cantilever electrical and thermal properties. The heater region dissipates more than 90% of the total cantilever power and its steady temperature was measured using laser Raman spectroscopy. Figure 3 shows the cantilever heater temperature and the cantilever electrical resistance as functions of the electrical power dissipated in the cantilever. The cantilever resistance increases with temperature as the carrier mobility of doped silicon decreases with temperature. The cantilever can be heated up to 600 °C, above which the diamond tip thermally oxidizes in ambient air. However below 600 °C, the cantilever could operate for long periods of time with negligible wear. All cantilevers in the cantilever array demonstrate similar electrical and thermal properties in the measured temperature range. A typical cantilever spring constant is about 0.8 N/m and resonant frequency is about 70 kHz.

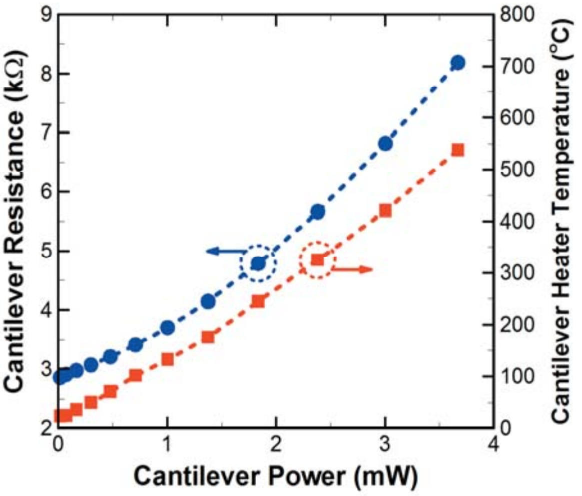

*Figure 3: Electrical and thermal characterization of the cantilever, measured using Raman spectroscopy.*

Figure 4 shows the wear test result of a UNCD tip in comparison with a silicon tip. To analyze tip wear, a UNCD tip and a silicon tip were scanned for 1.2 m at 10 µm/s speed on a single-crystal silicon grating with a feature height of 100 nm and a pitch of 3 µm. The applied load was held constant at 200 nN. The corresponding contact pressure will vary with tip radius; we estimate it ranges from 8 - 17 GPa based on Hertzian contact mechanics assuming tip the radius varying from 10 nm to 30 nm. Figure 4(a-b) shows SEM images of a UNCD tip before and after a 1.2 m scan, resulting in small change in tip radius from 28 nm to 36 nm. Figure 4(c-d) shows SEM images of a silicon tip before and after wear testing under the same testing conditions. A silicon tip is completely destroyed with severe fouling. These results demonstrate the utility of UNCD tips under harsh operating conditions, such as long scan distance, high tip load, and a hard substrate.

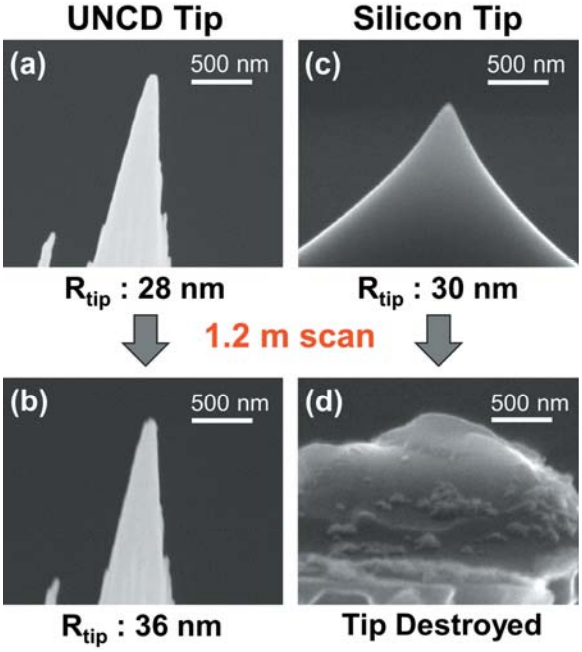

*Figure 4: Wear test comparison between the UNCD tip and the silicon tip. Both tips scanned for 1.2 m. The UNCD tip showed little wear while silicon tip got completely destroyed.*

## THERMAL TOPOGRAPHY IMAGING

With wear-resistant UNCD tips, heated AFM cantilevers can scan faster and for longer distances, meaning higher throughput and longevity for many applications. Here we demonstrate thermal topography imaging and thermal nanolithography using UNCD tip heated cantilevers.

Figure 5(a) shows the working mechanism of thermal topography imaging using a heated AFM cantilever. The heat flow from the cantilever to the touching substrate is inversely related to the gap between the cantilever and the substrate. By tracking changes in the cantilever power while the cantilever heater temperature is held constant, the substrate topography is measured. The quality of thermal imaging is quantified by topography sensitivity and noise limited resolution. Topography sensitivity is defined as the change in cantilever voltage for a unite change in topography. Noise limited resolution is defined as the smallest detectable change in topography and is calculated as the thermal signal noise divided by the topography sensitivity. For thermal imaging, the cantilever scanned 100 nm tall silicon gratings at 10 µm/s seed and at 100 nN applied force. The cantilever temperature was held at 410 °C and scanned in either contact mode or in intermittent contact mode at 75.67 kHz, which is the resonant frequency of the used cantilever.

Figure 5(b) shows the topography images of the substrate acquired from the conventional laser deflection signal and the thermal signal in both contact and intermittent contact modes. The thermal signals are inverted as the cantilever voltage changes inversely to the distance between the cantilever and the substrate. The topography sensitivity of the thermal signals is 0.079 ± 0.001 mV/nm via contact mode and 0.060 ± 0.001 mV/nm via intermittent contact mode imaging. The noise limited vertical resolution is 3.154 ± 0.062 nm via contact mode and 1.911 ± 0.036 nm via intermittent contact mode imaging. The acquired resolution is comparable to heated AFM cantilevers with silicon tips [2].

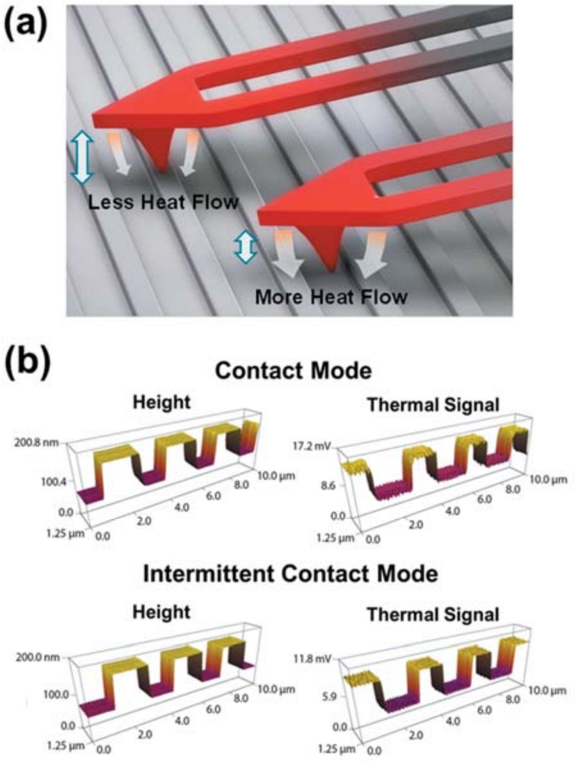

*Figure 5: (a) Schematic showing the concept of thermal topography imaging with heated AFM cantilevers. (b) Comparison of the laser deflection based measurement with the thermally derived signals while the cantilever was operated in both contact and intermittent contact mode.*

## THERMAL NANOLITHOGRAPHY

Heated AFM cantilevers can deposit polymer nanostructures [3, 6]. In thermal nanolithography, the polymer coated tip gets heated when it touches the substrate, causing the polymer to melt and flow onto the substrate. The polymer flow rate from tip to surface is sensitive to tip temperature, tip wettability, and tip geometry, since surface tension effects drive polymer flow [10].

Figure 6(a) shows an AFM image of 100 polyethylene nanostructures, deposited using a UNCD tip heated cantilever. The writing temperature was at 200 °C with a dwell time of 1 sec. Figure 6(b) shows a topographical profile image of one feature. The feature is approximately 100 nm tall and approximately 600 nm in diameter. These results are comparable to results obtained by depositing polymer features using a heated cantilever with a 1-µm-tall diamond-coated tip [6].

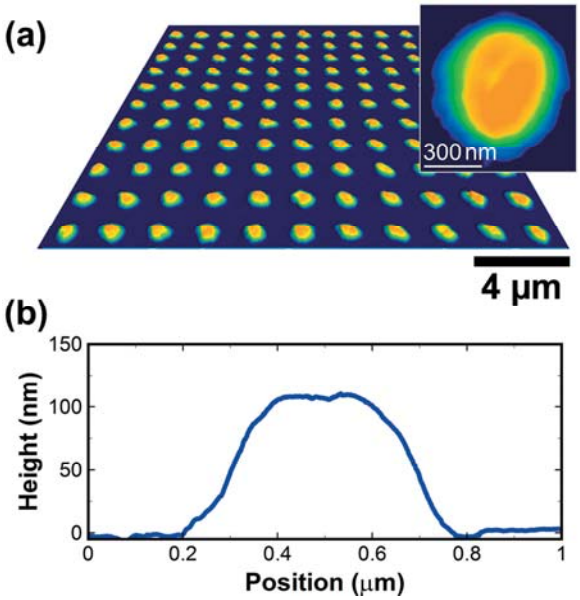

*Figure 6: (a) An AFM image of 100 polyethylene nanostructures deposited with heated UNCD tips. The inset shows the close-up of one nanostructure. (b) Topography profile of a nanostructure.*

## CONCLUSION

This paper reports heated AFM microcantilevers with ultrasharp UNCD tips, which were batch fabricated by a relatively simple direct mask RIE technique. The UNCD tip showed almost no wear after more than a meter of scanning while a silicon tip scanned under the same conditions was completely destroyed with much fouling. The UNCD tips were used for thermal topographic imaging with nanometer resolution, and also nanolithography of hundreds of polyethylene nanostructures at a writing speed of 1 Hz. The wear-resistant UNCD tips can expand the use of heated AFM cantilevers to imaging and tip-based nanofabrication under harsh conditions.

## REFERENCES

[1] P. Vettiger et al., "The "millipede" – nanotechnology entering data storage," *IEEE Transactions on Nanotechnology*, vol. 1, pp. 39-55, 2002.

[2] K. Park et al., "Topography imaging with a heated atomic force microscope cantilever in tapping mode," *Review of Scientific Instruments*, vol. 78, Apr 2007.

[3] B.A. Nelson et al., "Measuring material softening with nanoscale spatial resolution using heated silicon probes," *Review of Scientific Instruments*, vol. 78, p. 023702, 2007.

[4] D. Pires et al., "Nanoscale Three-Dimensional Patterning of Molecular Resists by Scanning Probes," *Science,* vol. 328, pp. 732-735, May 7 2010.

[5] H. Bhaskaran et al., "Ultralow nanoscale wear through atom-by-atom attrition in silicon containing diamond-like carbon," *Nature Nanotechnology*, vol. 5, pp. 181-185, 2010.

[6] P. C. Fletcher et al., "Wear-Resistant Diamond Nanoprobe Tips with Integrated Silicon Heater for Tip-Based Nanomanufacturing," *ACS Nano*, vol. 4, pp. 3338-3344, Jun 2010.

[7] A. V. Sumant et al., "Toward the Ultimate Tribological Interface: Surface Chemistry and Nanotribology of Ultrananocrystalline Diamond," *Advanced Materials*, vol. 17, pp. 1039-1045, 2005.

[8] A. Olbrich et al., "High aspect ratio all diamond tips formed by focused ion beam for conducting atomic force microscopy," *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures*, vol. 17, pp. 1570-1574, 1999.

[9] N. Moldovan et al., "Nanofabrication of sharp diamond tips by e-beam lithography and inductively coupled plasma reactive ion etching," *J. Vac. Sci. Technol. B*, vol. 27, pp. 3125-3131, 2009.

[10] J. R. Felts et al., "Nanometer-scale flow of molten polyethylene from a heated atomic force microscope tip," *Nanotechnology*, vol. 23, p. 215301, 2012.

## CONTACT

*William P. King, tel: +1-217-2443864, fax: +1 -217-2446534; wpk@illinois.edu