# Wear-Resistant Diamond Nanoprobe Tips with Integrated Silicon Heater for Tip-Based Nanomanufacturing

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**ABSTRACT** We report exceptional nanoscale wear and fouling resistance of ultrananocrystalline diamond (UNCD) tips integrated with doped silicon atomic force microscope (AFM) cantilevers. The resistively heated probe can reach temperatures above 600 °C. The batch fabrication process produces UNCD tips with radii as small as 15 nm, with average radius 50 nm across the entire wafer. Wear tests were performed on substrates of quartz, silicon carbide, silicon, or UNCD. Tips were scanned for more than 1 m at a scan speed of 25 μm $s^{-1}$ at temperatures ranging from 25 to 400 °C under loads up to 200 nN. Under these conditions, silicon tips are partially or completely destroyed, while the UNCD tips exhibit little or no wear, no signs of delamination, and exceptional fouling resistance. We demonstrate nanomanufacturing of more than 5000 polymer nanostructures with no deterioration in the tip.

**KEYWORDS:** atomic force microscope (AFM) · cantilever · ultrananocrystalline diamond (UNCD) · thermal dip-pen nanolithography (tDPN) · tip-based nanofabrication (TBN) · wear · nanotribology

Nanofabrication with scanning probes offers nanometer-scale feature resolution, immediate metrology of the written structures, and extraordinary flexibility in material choice. It has consequently been the subject of intense research.[1−11] A common requirement across all approaches to tip-based nanofabrication (TBN) is tip stability, which is essential for repeatable and consistent fabrication. Hard and/or chemically reactive substrates, long scan distances, high tip loads, and high temperatures all cause tip wear, deformation, and fouling,[12,13] thereby prohibiting the reproducibility required for manufacturing. This paper describes ultrananocrystalline diamond tips integrated into heated silicon atomic force microscope (AFM) cantilevers. These tips resist both wear and fouling under harsh conditions.

A number of techniques have been proposed for tip-based nanofabrication (TBN) such as depositing a material from a tip onto a surface[1−5] or using a tip to modify the mechanical,[6,7] electronic,[8] or chemical[9−11] properties of a surface. While most TBN techniques are slow (<1 μm $s^{-1}$), even for the fastest of them[10] at >1 mm $s^{-1}$, a probe array is required to reach reasonable manufacturing throughput. While some approaches have all the tips in an array write the same feature,[14] this greatly limits the complexity of the patterns formed. More versatile techniques use an array where each writing element can be independently addressed[15] such that each tip can generate an independent mechanical, thermal, or electrical field. Cantilevers with integrated heaters are particularly well-suited for TBN with large arrays as each individually addressed tip can write[4] and read[16] nanostructures in parallel.

TBN of nanoelectronics or lithographic masks requires the tip to scan long distances over hard surfaces such as silicon, silicon dioxide, quartz, or various metals. A number of tip materials and tip coatings have been suggested to reduce tip wear, including silicon dioxide tip encapsulation,[17] platinum silicide tips,[18] and various forms of carbon including diamond.[19−22] Diamond tips have the advantage of high stiffness and strength, low chemical reactivity and adhesion,[23] low friction coefficient,[24] and can be either electrically insulating or conducting if doped.[25] Typical diamond probes are fabricated by growing a thick diamond coating into a lithographically defined silicon wafer mold,[21] which is not well-suited to the electronic integration required for arrays of independently controlled tips. Alternatively, diamond thin films can be grown directly onto a silicon AFM tip,[22] but these methods usually

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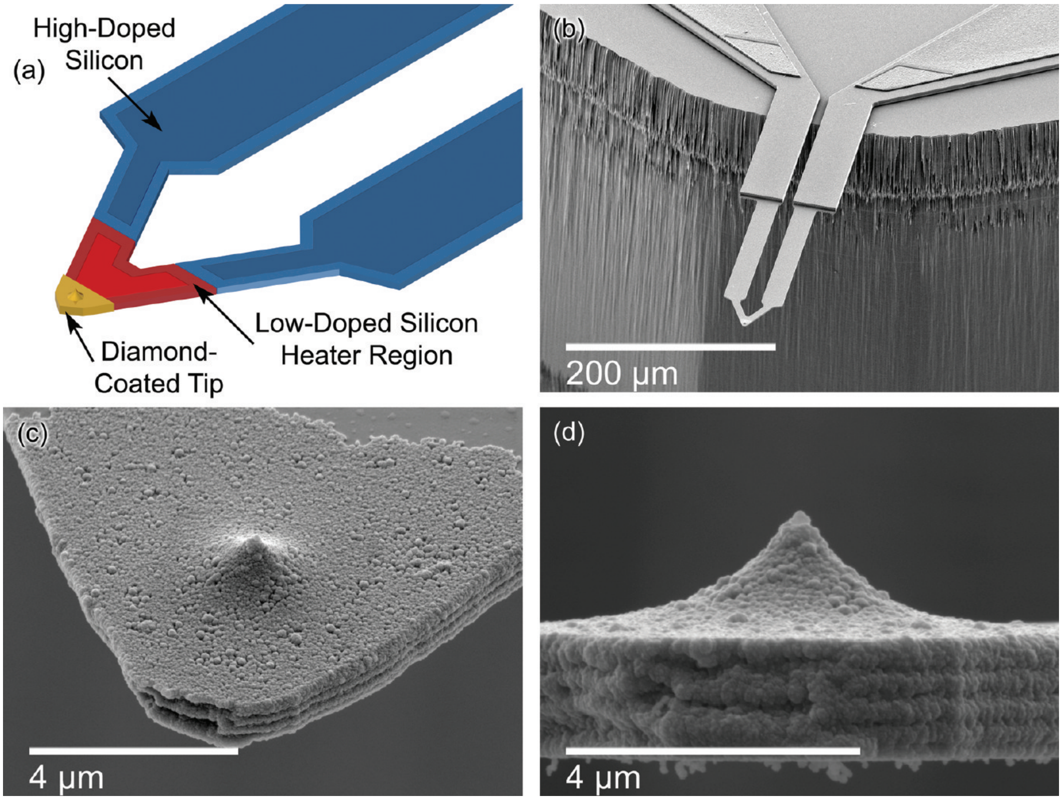

**Figure 1. Schematic (a) and SEM micrographs (b−d) of an ultrananocrystalline diamond tip integrated into a doped silicon heated AFM cantilever. The cantilevers were batch fabricated, with a typical tip radius of 50 nm. The supergrain structure produces multiple protrusions at the end of the tip with protruding asperities frequently <10 nm.**

produce highly stressed films with large grains leading to dull tips,[26] can delaminate,[27] or are highly graphitic leading to inferior chemical and mechanical properties.[28] Ultrananocrystalline diamond (UNCD) consists of 3−5 nm diameter crystalline grains of $sp^3$-bonded carbon, with 10% of the carbon located in high-energy, high-angle twist grain boundaries containing a mixture of locally $sp^3$- and $sp^2$-coordinated carbon.[24] Films of UNCD can be thin and conformal while having mechanical and chemical properties comparable to pure diamond. Recently, AFM tips fabricated entirely out of UNCD have been developed and shown to have far better wear resistance than commercial silicon nitride probes under room temperature testing conditions.[29]

This paper presents sharp UNCD-coated doped silicon tips with integrated heaters that have exceptional wear resistance under harsh conditions for scan distances greater than 1 m and that have dimensional stability during centimeter-scale tip-based nanofabrication.

Figure 1 shows the UNCD tip and its integration into the doped single-crystal silicon cantilever. The silicon cantilever legs are highly doped to carry current while the region near the cantilever tip is doped at a lower concentration to allow resistive heating.[30] Figure 2 shows the fabrication process. The radius of the sharpened silicon tip, typically ~10 nm, is increased by the UNCD coating, and therefore the coating must be as thin as possible while maintaining conformality, continuity, adhesion, and low roughness. After tip formation and cantilever doping, the 100 mm silicon wafer substrates were seeded with 5 nm diameter diamond nanoparticles by ultrasonication in a diamond nanoparticle colloidal suspension.[31] The UNCD was grown by hot-filament chemical vapor deposition. A protective silicon dioxide mask was patterned over the tip region, and the exposed UNCD was removed with an oxygen plasma etch, such that only the UNCD near the tip remained. Metal contacts and a backside etch completed the fabrication.

Figure 1 shows a fabricated cantilever and tip. The final devices had a 14 μm square film of UNCD centered on the tip that was approximately 40 nm thick. The UNCD coating was granular in appearance with supergrains typically 35 nm in diameter containing many smaller grains of UNCD. The size and morphology of these supergrains are directly related to the seeding process.[32] A small diamond supergrain protruding from the end of the probe tip would be optimal for the best sharpness, and this was observed in several cases. Each 4 in. diameter wafer yielded about 250 devices, where the average

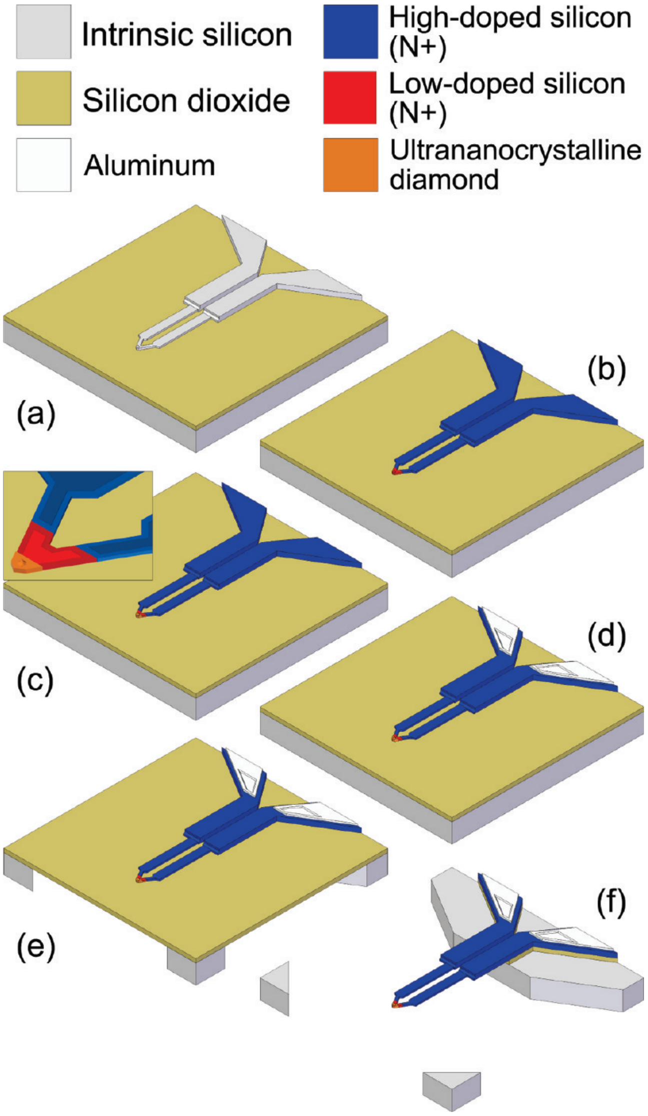

**Figure 2. Summary of fabrication steps. Fabrication begins with a silicon-on-insulator wafer. First, the anchor beams and tip cylinder are formed with inductively coupled plasma (ICP) deep reactive ion etching (DRIE). The tip is then sharpened with anisotropic wet etching and thermal oxidation. The final cantilever shape is formed in the device layer using ICP DRIE (a). The entire cantilever structure is low doped n-type with phosphorus, and subsequently, the cantilever legs are high doped n$^+$-type with phosphorus (b). The ultrananocrystalline diamond (UNCD) is conformally grown on all silicon surfaces and then etched away with oxygen plasma, leaving a protected UNCD window around the AFM tip (c). The cantilever is coated with a silicon dioxide insulating layer, vias to the highly doped silicon are exposed, and aluminum traces are deposited for electronic connection to the highly doped silicon (d). The device handle is created with backside etching through 500 μm of silicon, using the buried oxide layer as an etch stop (e). Finally, the sacrificial oxide layer was removed, releasing the heated cantilever (f).**

overall tip radius was 50 nm according to SEM, with supergrain protrusions of radius 5−15 nm.

The electrical, thermal, and mechanical properties of the cantilever were characterized using established techniques.[30] The cantilever can be heated to above 800 °C; however, diamond burns above 600 °C in air. Indeed, the diamond completely oxidized and was removed after heating to 750 °C (see Supporting Information). The cantilever spring constant was 0.15−6 N m$^{-1}$ depending on the cantilever thickness, which varied from 0.75 to 1.5 μm depending on location on the wafer.

The UNCD-coated AFM tips were tested for wear resistance in comparison to uncoated silicon tips. While previous reports show the durability of diamond-coated silicon tips,[27,33] the present work uses significantly harsher test conditions including the opportunity to perform experiments at elevated temperature. The tips were imaged in an SEM before and after each test, and the wear was monitored *in situ* during the experiment by monitoring the evolution of the tip−substrate pull-off force[34] (see Supporting Information). We wrote a custom IGOR program to raster the tip on the substrate and measure the tip−substrate pull-off force after every scan. Each wear test took an average of 17 h to complete; subsequently, the biggest challenge was overcoming drift in the system over such a long time period which sometimes caused the AFM laser to drift off the cantilever and cause an error in the program.

The tip contact force, cantilever temperature, and substrate material varied between experiments, but in all tests the tip scanned a total of 1.28 m at a scan speed 25 μm s$^{-1}$. Silicon and UNCD tips were tested with a tip contact force varying from 10 to 200 nN, where 200 nN is the maximum tip force possible in our apparatus. The experiments tested cantilever tips with self-heating temperatures varying from 25 to 400 °C. Most tests were performed on either polished silicon carbide (SiC) or quartz substrates, although some tests were performed on polished single-crystal silicon, or UNCD films with 10 nm rms roughness. The relative humidity was not controlled but was recorded for each test. For the present experiments, there was no systematic dependence of wear on humidity, which could be attributed to the elevated cantilever temperature or the specific tip−substrate chemistries.

The UNCD-coated tips were remarkably durable under all wear test conditions and consistently outperformed the silicon tips. Over all of the experiments, the diamond-coated tips had an average wear rate of $\sim 1 \times 10^{-16}$ m$^3$ N$^{-1}$ m$^{-1}$, whereas the silicon tips had a 100-fold higher rate of $\sim 1 \times 10^{-14}$ m$^3$ N$^{-1}$ m$^{-1}$. Figure 3 shows example images before and after wear testing, and Table 1 shows complete wear testing results without tip micrographs. On the silicon substrate, the silicon tips experienced moderate wear while the UNCD tips were unaffected. No deformation of the silicon substrate was observed after the UNCD tip wear test. The polished SiC substrate produced the least tip wear for both silicon and UNCD tips. The quartz substrate and the UNCD substrate destroyed the silicon tips while only

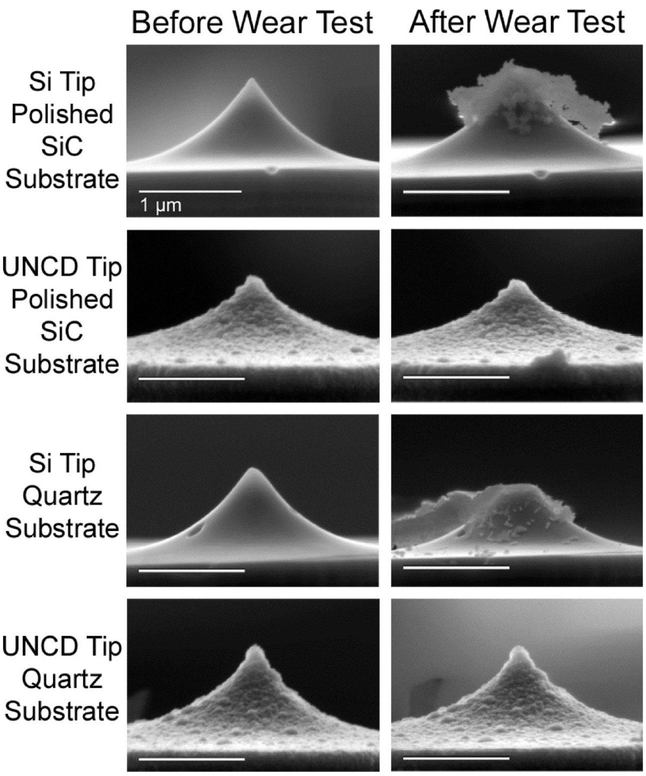

**Figure 3. Representative SEM micrographs of tip wear. Before and after micrographs read from left to right. From top to bottom: silicon tip at 400 °C with a force of 200 nN on polished SiC; UNCD tip at 400 °C with a force of 200 nN on polished SiC; silicon tip at 400 °C with a force of 200 nN on quartz; UNCD tip at 400 °C with a force of 200 nN on quartz. The UNCD tips showed little or no wear and little or no debris accumulation. The silicon tips showed significant wear and debris accumulation.**

slightly wearing the UNCD tips. Importantly, we observe no signs of the delamination commonly found with commercial diamond-coated probes when used even for metrology and not the harsh conditions relevant to TBN investigated here.

The primary wear mechanism for the UNCD tips is gradual atom-by-atom attrition of the sliding surface. Importantly, we do not observe a significant increase in wear rate at elevated temperatures that would be expected in the model developed by Gotsmann and Lantz.[34] Figure 4 shows bright-field TEM images of a tip before and after a wear test. While volume has clearly been removed, the unworn material appears unaffected, showing no signs of graphitization. Moreover, the selected area diffraction patterns were unchanged with wear, with no evidence of graphitization.

In addition to wear, tip performance can be degraded by accumulation of debris, and indeed, tip fouling is the most common mechanism for probe failure in typical AFM operation. The UNCD probes resisted such fouling on most substrates, except for the UNCD substrate, where there was slight transfer. The silicon tips accumulated measurable debris on all substrates; in some cases, the amount was significant compared to the tip size, as shown in Figure 3. The antifouling characteristics of the diamond tip can be attributed to the low surface energy as well as the chemical stability of the diamond. Supporting Information shows representative *in situ* tip pull-off force measurements, the complete series of before and after SEM tip images for all experiments, and a description of TEM measurements including selected area diffraction images.

To demonstrate the stability of a UNCD-coated heated probe for tip-based nanofabrication, we conducted an extended TBN experiment with a single tip. Figure 5 shows thermal deposition of polymer from a tip that is heated or cooled to modulate nanostructure writing. The polymer was poly(3-dodecylthiophene) (PDDT), a semiconducting polymer,[35] and the substrate was polished silicon. The cantilever temperature was switched between room temperature and 120 °C while the tip scanned continuously at 1 μm $s^{-1}$, producing 300 nm wide polymer nanostructures with alternating lengths of 2.5 and 1.5 μm and spacing of 1.5 μm. In total, 5400 nanostructures were written and the total scan distance was 1.89 cm. The tip was cleaned after every 1000 lines for SEM imaging, although at no point was the polymer noticeably depleted. Figure 5 shows the number of features written and total scan distance. Figure 5 also shows images of polymer nanostructures numbers

TABLE 1. **Silicon and UNCD-Coated AFM Cantilever Wear Testing Results**

| tip type | surface | force (nN) | temp (°C) | radius before (nm) | radius after (nm) | tip wear |
|---|---|---|---|---|---|---|
| silicon | polished Si | 200 | 400 | 32 | 42 | moderate |
| UNCD | polished Si | 200 | 400 | 47 | 49 | slight |
| silicon | polished SiC | 10 | 25 | 25 | 48 | moderate |
| silicon | polished SiC | 200 | 25 | 25.5 | 65 | moderate |
| silicon | polished SiC | 200 | 200 | 50 | 57 | moderate |
| silicon | polished SiC | 200 | 400 | 30.5 | N/A | extreme |
| UNCD | polished SiC | 10 | 25 | 32 | 34 | slight |
| UNCD | polished SiC | 200 | 25 | 27 | 27.5 | slight |
| UNCD | polished SiC | 200 | 200 | 61 | 61 | none |
| UNCD | polished SiC | 200 | 400 | 67 | 67.5 | slight |
| silicon | quartz | 10 | 25 | 22 | N/A | extreme |
| silicon | quartz | 200 | 25 | 42 | N/A | extreme |
| silicon | quartz | 200 | 200 | 35 | 138 | extreme |
| silicon | quartz | 200 | 400 | 66.5 | 187 | extreme |
| UNCD | quartz | 10 | 25 | 46 | 46 | none |
| UNCD | quartz | 200 | 25 | 44 | 53 | moderate |
| UNCD | quartz | 200 | 200 | 35 | 43 | slight |
| UNCD | quartz | 200 | 400 | 89 | 90 | slight |
| silicon | UNCD | 200 | 400 | 65 | 245 | extreme |
| UNCD | UNCD | 200 | 400 | 26 | 73 | moderate |

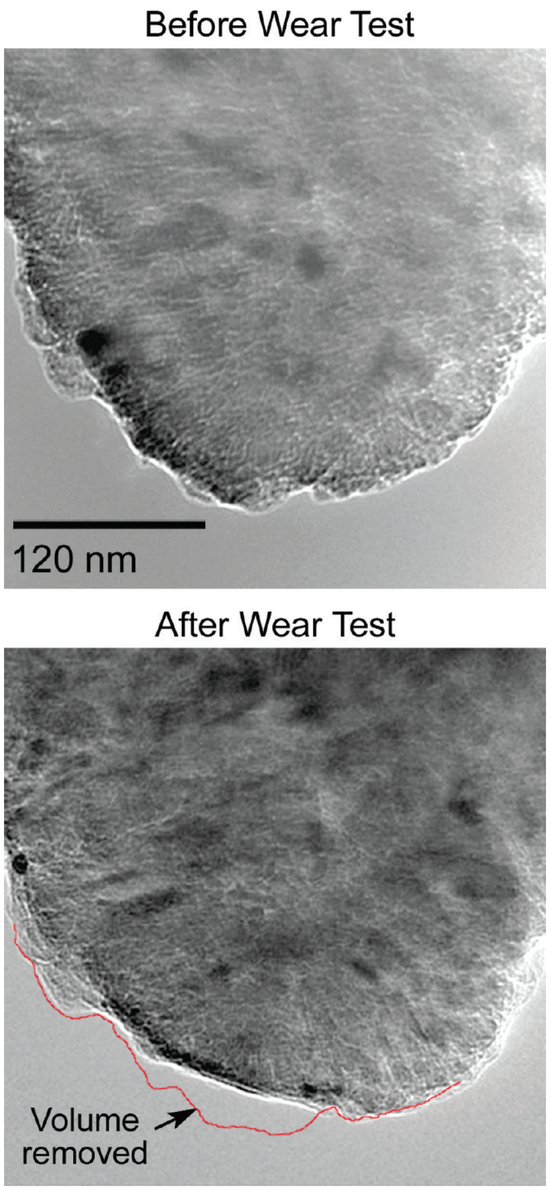

Figure 4. Transmission electron microscope (TEM) images of a UNCD tip before and after wear testing on a quartz substrate at 400 °C, load force 200 nN, and scan distance 1.28 m. The expected morphology of high-quality UNCD is observed, showing <5 nm grains clustered in supergrain structures.

1060−1080, numbers 5380−5400, and images of the tip at the corresponding point in the experiment.

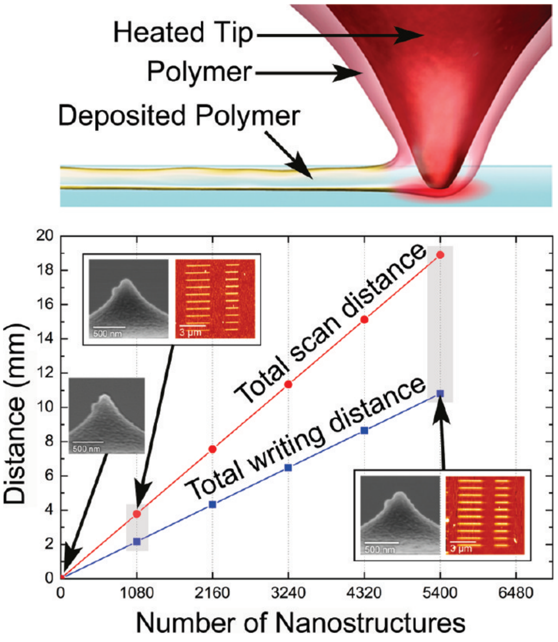

Figure 5. Thermal deposition of polymer nanostructures using a heated UNCD cantilever tip; 5400 polymer nanostructures were written at at heater temperature of 120 °C and consisted of alternating 2.5 and 1.5 μm lines. The plot shows both the writing distance and the total scan distance for one tip. Insets show the tip at different times during writing, as well as several nanostructures written at various times.

The stability of the tip shape allowed nearly identical polymer nanostructures to be written over the entire experiment.

By harnessing the wear resistance and stability of diamond with silicon electronic integration, it would be possible to make and use massive arrays of robust and independently controlled nanoprobe tips. Such arrays would be ideal for nanofabrication. Consider an array of $10^6$ probe tips writing 25 nm structures onto a 100 mm wafer. To fill this wafer, each tip would travel 1.26 m and, assuming a 10% fill, each tip would write for only 12.6 cm. Diamond probe tips can easily travel such distances with exceptional stability, overcoming the most significant challenges to tip-based nanofabrication.

## METHODS

**Wear Testing:** The cantilever mechanical characteristics were measured in an Asylum MFP-3D AFM, which was also used for all wear tests. Before each test, we measured cantilever stiffness using the thermal method, which relies on the equipartition principle from classical thermodynamics to equate the mechanical fluctuations of the cantilever with its thermal energy.[36] The cantilever fundamental frequency was measured directly using the AFM piezo-actuator and optical laser. The total scan distance of 1.28 m corresponded to 1000 scans on a 1 μm × 1 μm area. Each scan area consisted of 1024 line scans, including retrace paths, and each line scan was 1.25 μm long, including excess tip travel outside the scan area. After every area scan, we measured the tip−substrate pull-off force (see Supporting Information). We wrote a custom IGOR program to raster the tip on the substrate and measure the tip−substrate pull-off force after every scan. Each wear test took an average of 17 h to complete.

**Selected Area Diffraction:** We investigated the material properties of the UNCD and performed selected area diffraction on the samples using a JEOL 2010F field-emission TEM at 200 kV accelerating voltage.

**Loading and Cleaning of the UNCD Tip:** The cantilever was loaded with polymer by inserting the probe tip into a drop of chloroform containing PDDT. The chloroform would then evaporate, leaving a large amount of polymer on the tip. The tip was heated while in contact with the substrate and scanned to remove the bulk of the polymer, leaving behind a thin layer of PDDT around the tip to be used for patterning. We cleaned the tip using chloroform and a low power 100 W oxygen plasma after every 1000 lines for SEM imaging. Although oxygen plasmas etch UNCD, the power level of the plasma was low enough such that appreciable etching did not occur. Additionally, the plasma served to improve the adhesion of PDDT onto the UNCD coating for polymer loading by removing the hydrogen and oxygen atoms terminating the dangling carbon bonds.

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*Supporting Information Available:* Electrical and thermal characterization of heated cantilevers. Images showing removal of UNCD from heated cantilever tip through oxidation and self-heating to high temperatures. TEM images of UNCD-coated tip before and after wear testing. Wear test results for polished silicon, polished silicon carbide, amorphous quartz, and ultrananocrystalline diamond. This material is available free of charge *via* the Internet at http://pubs.acs.org.

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