

ADVANCED FUNCTIONAL MATERIALS www.afm-iournal.de

Wear-Resistant Nanoscale Silicon Carbide Tips for Scanning Probe Applications

Mark A. Lantz,* Bernd Gotsmann, Papot Jaroenapibal, Tevis D. B. Jacobs, Sean D. O'Connor, Kumar Sridharan, and Robert W. Carpick

The search for hard materials to extend the working life of sharp tools is an age-old problem. In recent history, sharp tools must also often withstand high temperatures and harsh chemical environments. Nanotechnology extends this quest to tools such as scanning probe tips that must be sharp on the nanoscale, but still very physically robust. Unfortunately, this combination is inherently contradictory, as mechanically strong, chemically inert materials tend to be difficult to fabricate with nanoscale fidelity. Here a novel process is described, whereby the surfaces of pre-existing, nanoscale Si tips are exposed to carbon ions and then annealed, to form a strong silicon carbide (SiC) layer. The nanoscale sharpness is largely preserved and the tips exhibit a wear resistance that is orders of magnitude greater than that of conventional silicon tips and at least 100-fold higher than that of monolithic, SiO-doped diamond-like-carbon (DLC) tips. The wear is well-described by an atom-by-atom wear model, from which kinetic parameters are extracted that enable the prediction of the long-time scale reliability of the tips.

1. Introduction

New scientific and technological avenues are continually enabled by atomic force microscopy (AFM). The nanoscale interaction between an AFM tip and a sample enables imaging, material characterization, and now, manufacturing on a nanometer scale. Nanomanufacturing using AFM probes is an important subfield in the emerging broad area of nanomanufacturing. Such techniques can be used to produce nanostructures or nanopatterns on surfaces, for applications including biosensors and

Dr. M. A. Lantz, Dr. B. Gotsmann IBM Research-Zurich, Säumerstrasse 4, Rüschlikon, CH8803 Switzerland E-mail: mla@zurich.ibm.com Dr. P. Jaroenapibal, Prof. R. W. Carpick Department of Mechanical Engineering and Applied Mechanics University of Pennsylvania 220 S. 33rd St., Philadelphia, PA 19104, USA T. D. B. Jacobs Department of Materials Science and Engineering University of Pennsylvania 3231 Walnut St., Philadelphia, PA 19104, USA S. D. O'Connor, Prof. K. Sridharan Deptartment of Engineering Physics University of Wisconsin 1500 Engineering Drive, Madison, WI 53706, USA

DOI: 10.1002/adfm.201102383



data storage, and can be used for nanolithography for high-resolution masks or templates.^[1–13] One of the most developed nanomanufacturing techniques is based on thermomechanical probes, which have been applied to nanoscale lithography,^[2–4] data storage^[14–18] and thermal imaging.^[19] All of these developments critically rely on the durability and stability of the tip itself. The tip can be subjected to harsh chemical environments, elevated temperatures and high mechanical and thermal stresses, which can lead to dramatic wear.^[20,21]

Most tip-based nanofabrication (TBN) techniques use Si tips, which can readily be fabricated with radii of <10 nm. Unfortunately, the wear rates of Si tips are high, particularly in ambient conditions where water vapor accelerates wear tribochemically.^[20] One approach to improve the wear resistance of Si tips is to coat them

with harder materials;^[22,23] however, this usually blunts the tip and the wear robustness may be limited by the adhesion of the coating to the Si rather than its hardness. Another approach is to mold tips from a hard material such as diamond-like carbon (DLC).^[24–26] Unfortunately, such tips may be unsuitable for nanofabrication applications that require elevated tip temperatures because DLC can destabilize between 250 to 400 °C by losing hydrogen and eventual rehybridization of carbon from sp³ to sp² bonding.^[27]

The ideal tip material for thermomechanical nanofabrication should have a high hardness, good temperature stability and chemical inertness and a high thermal conductivity. Electrical conductivity is also advantageous to prevent charging and to enable current- and field-based TBN methods. Silicon carbide (SiC) fulfills all of these criteria. It has a melting point of 2830 °C,^[28] it is highly chemically inert, it has a high thermal conductivity (3.6^[29] versus 1.56 W cm⁻¹ °C⁻¹ for Si^[30]) and it is among the hardest materials known (up to 50 GPa for nanocrystalline forms^[31]). However, it is challenging to micromachine structures with nanoscale fidelity from SiC films.^[32,33] Moreover, the processes used to fabricate nanometer-scale tips from Si cannot readily be applied to the manufacture of SiC tips.

Here we describe a process for forming nanoscale SiC layers on the surface of tips of prefabricated Si AFM cantilevers by depositing and implanting carbon into sharp single-crystalline Si tips, followed by annealing to achieve in situ conversion





to SiC at the tip surface with little change in the tip size and shape. Wear tests demonstrate a nanoscale wear resistance several orders of magnitude higher than that of Si tips, and at least two orders of magnitude higher than that of tips fabricated monolithically from SiO-doped DLC (a material with ultralow wear at the macroscale).

2. Silicon Carbide Formation

Carbon-ion implantation and deposition were performed on Si wafers and Si cantilevers with sharp tips and integrated tip heaters^[34] using a plasma immersion ion implantation and deposition (PIIID) process^[35–37] with a CH₄ precursor gas. PIIID facilitates the uniform surface treatment of stationary 3D parts.^[35] It is thus well-suited to processing high–aspect-ratio AFM tips. In PIIID, ions are generated in a plasma, and the sample is pulsebiased to a negative voltage, accelerating ions into the sample at near-normal incidence. Between pulses, species from the plasma condense on the surface, resulting in the co-deposition of material. The CH₄ precursor gas results in the deposition of a carbon layer on the sample surface, as well as in the implantation of a variety of ion species, including CH₄⁺, CH₃⁺, CH₂⁺ CH⁺ and C⁺,^[38] which dissociate, resulting in an implanted, C-rich layer near the surface and a deeper hydrogen-rich region.

The PIIID-treated cantilevers used in this work were designed for thermomechanical data storage and lithography and had built-in resistive heaters above the tip.^[34] To study the effect of the implantation on the tip shape, high-resolution scanning and transmission electron microscopy (SEM, TEM) images were obtained before and after implantation. All of the experiments used a PIIID process time of 60 min. An acceleration voltage of -10 kV applied to the tip at a pressure of 1.6 mTorr resulted in a significant increase in tip radius, due to sputtering effects and carbon deposition. This blunting was reduced by decreasing the acceleration voltage to -5 kV and the pressure to 0.5 mTorr. Figure 1a,b shows high-resolution TEM images of a typical tip before and after this latter process, which produced an average increase of the tip radius of only 2 nm. The PIIID process caused the resistance of the built-in heaters roughly to double, and induced stress in the top surface of the cantilever that led to a bending of a few degrees. The original

resistance was recovered by annealing the built-in heaters at ≈ 600 °C for a few seconds. The change in resistance likely resulted from the implantation of hydrogen ions that were subsequently driven out with annealing. Such effects can readily be avoided by exposing only the tips to the PIIID process during the cantilever fabrication process.

PIIID-treated Si can be converted to SiC by annealing at temperatures above 800 °C.^[38] To understand and optimize the conversion process, we performed PIIID on Si wafers that were then cleaved into dies and separately annealed under vacuum at temperatures ranging from 800 to 1200 °C in a rapid thermal annealer (RTA) for durations of 10 s, 100 s and 1000 s.

Cross-sectional TEM images of the Si dies treated with a 5 kV, 0.6 mTorr PIIID process revealed a distinct layer on the surface of ~20 nm thickness (**Figure 2**a). A 10 kV, 1.5 mTorr process resulted in a similar layer, ~40 nm thick. TEM-based energy dispersion spectroscopy (EDS) scans showed that these layers consisted of >96 at% carbon. With the 10 kV process, an implanted C-rich layer was clearly observed ~40 nm below the surface. Simulations using "stopping and range of ions in matter" (SRIM) (http://www.srim.org/) predicted a similar implantation depth.

The annealing step accelerates interdiffusion of carbon and silicon atoms and promotes the conversion of the surface region into silicon carbide. EDS analysis of the samples annealed at 1100 °C indeed showed that Si diffused outward to the surface and C diffused into the Si substrate. After annealing at 1100 °C for 100 s, the thickness of the surface layer was reduced to \approx 16 nm (Figure 2b). Lattice resolution was observed in the Si substrate, whereas the 16 nm surface layer appeared disordered. High-resolution images of this surface layer revealed some evidence of small nanocrystalline regions (Figure 2c).

The treated silicon wafers were also studied using absorption Fourier transform infrared (FTIR) spectroscopy. SiC exhibits an IR absorption peak at 795 cm⁻¹, attributed to transverse optical (TO) phonons^[38,39] (**Figure 3**a). Prior to annealing, no features were observed in the FTIR spectra in this region. In the annealed samples, we observed a broad FTIR spectroscopy peak at \approx 795 cm⁻¹, which increased in height and sharpness with increasing annealing temperature and time, and which exhibited a maximum height for the sample annealed at 1100 °C for 100 s (see Supporting Information). This peak is a clear indication



Figure 1. a–c) TEM images of tip apices: typical tip prior to implantation, with tip radius R = 5 nm (a); another typical tip after the PIIID process, R = 7 nm (b); the tip shown in (b) after annealing at 1100 °C for 100 s, R = 6.5 nm (c).

www.MaterialsViews.com

FULL PAPER



Figure 2. a,b) TEM images of cross sections taken from Si die: after the PIIID process (5 keV, 0.6 mTorr, 60 min) (a) and after vacuum annealing at 1100 °C for 100 s (b). The Pt capping layer in the bottom part of the images was deposited as part of the TEM sample preparation process. The carbonrich surface layer had an initial thickness of \approx 20 nm (a) and was reduced to \approx 15 nm after annealing (b). c) High-resolution image of a second sample showing evidence of crystalline regions (red circles) in the C-rich surface region after annealing at 1100 °C for 100 s.

of SiC formation. The activation energy for the conversion into SiC was estimated to be \approx 2.0 eV using a threshold absorption peak height of 0.02, as shown in Figure 3b. A wide range of annealing temperatures and activation energies have been reported in the literature for various SiC fabrication methods.^[40–43] Our values are consistent with values reported for conditions that, in addition to vacancy diffusion, also require SiC-bond formation and crystal nucleation.^[40–43]

X-ray photoelectron spectroscopy (XPS) was used to confirm the formation of SiC at the surface. While transmission FTIR spectroscopy is sensitive to changes throughout the sample, XPS is sensitive only to the surface, up to depths of a few nanometers. Figure 3c shows the C 1s XPS spectra of an as-implanted sample and five samples annealed for 100 s at temperatures ranging from 800 to 1200 °C. The films annealed up to 1000 °C exhibit a broad peak in the range of 283 to 286 eV, reflecting the various binding configurations of the deposited carbon. At higher temperatures, a clear separation into two peaks is observed. The lower binding energy peak at 283 eV is expected for SiC.^[44–46] The contribution at \approx 285 eV was assigned to C–C



Figure 3. a) FTIR spectra obtained for the C-implanted silicon dies. The formation of SiC is indicated by the characteristic peak at 795 cm⁻¹. Annealing at 1100 °C for 100 s yields optimal results. A linear background was subtracted from each spectrum. Only data for annealing times of 100 s are shown, with offsets assigned for clarity (see Figure S2, Supporting Information, for spectra at longer and shorter annealing times). b) A kinetic study of the conversion of C and Si into SiC, based on the height of the FTIR spectroscopy absorption peaks exceeding a threshold of 0.02 in the units of Figure 3a (see Figure S2, Supporting Information, for corresponding peak heights). c) XPS spectra of the same samples (only the 100 s anneals are shown for clarity): a linear background was subtracted from each spectra, with offsets assigned for clarity. The formation of SiC is indicated by the characteristic peak at 283 eV. Carbon–carbon bonds appear at 285 eV, and, at higher binding energies, counts due to oxidized carbon are detected. d) The atomic concentration ratio of carbon to silicon, extracted from the XPS spectra of C and Si, as a function of annealing temperature for 100 s anneals is shown.



bonds. The Si 2p XPS peak also exhibits a behavior consistent with the formation of SiC (see Supporting Information). The XPS data indicate a maximum conversion to SiC in the surface region for annealing temperatures of 1100 to 1150 $^{\circ}$ C.

XPS can also be used to determine the surface composition. The C-to-Si atomic ratio initially was \approx 5. With annealing, the ratio continuously decreased and reached a value of 1.0 at 1100 °C and 0.9 at 1150 °C for the 100 s anneal time (see Figure 2d). The detection of SiC at the surface by XPS indicates that out-diffusion of Si, inward-diffusion of C, volatilization of C, or combinations thereof, led to the conversion of the initially C-rich surface layer into SiC for annealing at 1100 °C for 100 s.

Based on the TEM, EDS, FTIR spectroscopy, and XPS data, we conclude that the 5 kV PIIID process followed by annealing for 100 s at 1100 °C resulted in the formation of a SiC surface layer. The layer was mostly amorphous, with some nanocrystal-line regions.

This optimized annealing process of 100 s at 1100 °C was also applied to the tip shown in Figure 1b using the built-in tip heater under vacuum ($P = 10^{-7}$ mbar). Although there was some uncertainty in the tip temperature during annealing with the integrated heaters, the tip images also revealed a slight reduction in the thickness of the surface layer after annealing, and a lighter contrast over the outermost \approx 30 nm of the tip apex, consistent with the result for amorphous SiC found for the implanted and annealed die. After annealing, the radius of the tip apex was approximately 6.5 nm (Figure 1c), as compared to \approx 7 nm prior to annealing. In general, we observed a slight decrease in tip radius after annealing, consistent with the reduction in surface-layer thickness observed for the silicon die. However, this change was at the resolution limit of the TEM images.

3. Wear Testing

Nanoscale wear experiments were performed using two counter surfaces: a 100 nm-thick film of cross-linked polyaryletherketone (PAEK) polymer spun-cast onto Si, and a 100 nm thermal SiO₂ layer on Si. The polymer serves as model system for data storage^[47] and lithography applications,^[2] whereas SiO₂, which is much more abrasive, serves as a model surface for CMOS (complementary metal-oxide-semiconductor) metrology applications.^[26]

The wear experiments were performed using a custom-built AFM housed in a vacuum chamber for environmental control. The wear experiments involved raster-scanning tips over a $30 \,\mu\text{m} \times 6 \,\mu\text{m}$ area of the sample in constant-height mode (i.e., without fast *z*-feedback), and without the acquisition of image data (to enable high scanning speeds). To minimize load variations, the tip was scanned parallel to the predetermined plane of the sample surface. The low roughness of the two surfaces had a negligible influence on variations in the load (in total, <3 nN). The fast-scan axis (mean velocity of 1.5 mm s⁻¹) was driven sinusoidally to avoid ringing of the scanner. Normal-force versus displacement curves were acquired after every 6.2 cm of sliding to provide slow feedback control of the load and to monitor the state of the tip as described below. After every 6.2 cm of sliding, a reduced scan velocity was used to acquire an image of the area in which fast sliding had been performed to monitor the state

of the counter surface. In all of the experiments, no significant changes in surface topography were detected.

During the wear experiments, the tip-sample pull-off force provided a measure of the tip geometry. In general, the pull-off force depends on both the tip and sample materials and the tip geometry. For the flat punch geometry typical of nanoscale wear tests, the pull-off force, F_{adh} , is assumed to be proportional to the radius of the flat end, *a*, viz. $F_{adh} = k_{adh}a$, which is appropriate in the limit of small contacts with low adhesion.^[20]

Figure 4 compares the wear performance of a Si tip and a SiC tip sliding on the polymer sample for 100 m (3.33×10^6 linear sliding cycles, 30 µm long). The SiC wear test was performed under relatively harsh conditions, namely ambient humidity (\approx 45% relative humidity (RH)) and 10 nN of applied load. The SiC tip was prepared using the 5 kV PIIID process and annealed at 1100 °C in vacuum (<10⁻⁶ mbar) for an extended time of 200 s to ensure the formation of SiC, due to the tip-temperature uncertainty. A Si tip under identical conditions produces so much wear that continuous tip-radius sampling via adhesion is difficult because of wear debris generation. Therefore, Figure 4 shows a Si-tip experiment performed under less harsh conditions of <4% RH and 2.5 nN load.

The SEM images of the Si tip before (Figure 4a) and after (Figure 4b) the wear test reveal a worn volume of approximately 25 200 nm³, whereas wear was not detectable for the SiC tip (Figure 4c,d). However, the tip-surface adhesion tests (Figure 4e) indicate that some wear had occurred. We estimate an upper bound of the wear volume^[20] (also see Supporting Information) of 450 nm³, >50 times less than that for the Si tip, despite the significantly harsher conditions.

Figure 5 shows a comparison of the wear performance of a Si tip and a SiC tip (prepared using the same process as the tip shown in Figure 4c) sliding on SiO₂ for 40 m (1.33 \times 10⁶ linear sliding cycles, 30 µm long), under ambient conditions (≈45% RH) with a 10 nN applied load. The SEM images of the Si tip before (Figure 5a) and after (Figure 5b) the wear test reveal a worn volume of approximately 5.8×10^6 nm³, much higher than that for sliding on PAEK at low humidity. Figure 5c,d shows low-magnification TEM images of the SiC tip before and after the wear test, respectively, and Figure 5e,f shows high-magnification images before and after the wear test, respectively. Comparing Figure 5e and f, we estimated a wear volume of $\approx 700 \text{ nm}^3$, approximately four orders of magnitude lower than for the Si tip. Figure 5g shows a comparison of the adhesion data acquired during the two 40 m wear tests, which also demonstrates a strikingly lower wear rate for SiC.

Our previous work with monolithic, SiO-doped DLC tips sliding on SiO₂ for ~2 m with applied loads of ~1–17.5 nN^[26] yielded wear volumes of 2000 to 10 000 nm³. Compared with that work, the thin SiC layer exhibits an approximately tenfold lower wear volume than the monolithic DLC tips, despite being subjected to a 20-times longer sliding distance.

4. Wear Modeling and Discussion

Several nanoscale wear studies have reported that wear rates (i.e., wear volume divided by sliding distance) depend on the tip geometry and load in a non-linear manner. To explain this

Materia

www.MaterialsViews.com





Figure 4. a–d) Comparison of the wear performance of Si and SiC tips sliding on a cross-linked polymer sample. SEM images: before (a) and after the wear test (b) for the Si tip; and before (c) and after (d) the wear test for the SIC tip. e) Adhesion data collected during the wear tests of the Si tip (red) and the SiC tip (black squares) and a fit of the wear model to the SiC wear data (blue). The adhesion data provide a quantitative measure of the increase in radius of the tip apex as the wear progresses (see Supporting Information).

behavior, a thermally activated wear model which assumes a progression of single bond-breaking events that are thermally activated, was recently proposed.^[20,48] The activation energy, E_a , of the dominant bond-breaking process is the decisive parameter

Figure 5. a-f) Comparison of the wear performances of Si and SiC tips sliding on a SiO₂ sample: SEM images of the Si tip, before (a) and after (b) the wear test; TEM images of the SiC tip before (c) and after the wear test (d); and high-resolution TEM images of the SiC tip apex before (e) and after (f) the wear test. g) Plot of adhesion data collected during the wear tests of the Si tip (red) and the SiC tip (black). The inset shows a fit of the wear model to the SiC wear data.

controlling the transition rate. In the model, it is assumed that the applied frictional shear stress, τ , reduces the original activation barrier, E_0 , to an effective barrier: $E_a = E_0 - V_{act}\tau$, where V_{act} is a suitable activation volume. Taking into account that the





shear stress is usually found to have a small dependence on the contact pressure *p*, viz. $\tau = \tau_0 + \zeta p$, only two material-dependent parameters govern the wear process: the effective activation energy, $E_{\rm eff} = E_0 - V_{\rm act}\tau_0$, and the effective activation volume, $V_{\rm eff} = V_{\rm act}\zeta$. Here, ζ and τ_0 are constants.

The model describes well the wear of the Si and the SiOdoped DLC tips mentioned earlier. Here, we have applied it to the SiC-tip wear data and obtained good fits to the data (blue lines in Figure 4 and 5). These fits enable the wear resistance of the various tip materials to be quantitatively compared by predicting the amount of wear that would take place if the load and tip geometry were the same. The model predicts that, for tips sliding on PAEK polymer under identical conditions with an initial tip shape as that shown in Figure 4, a SiC tip can slide 1000 times further than a Si tip before the same wear volume is produced. Comparing SiC and SiO-doped DLC tips sliding over SiO₂, we found that the SiC tip can slide 300 times further. We note, however, that in the case of DLC tips, a direct comparison is more difficult due to differences in the experimental conditions used, as discussed in the Supporting Information.

We could not determine the bond energies and activation volumes directly, because the friction and the bond-breaking properties are interdependent. The effective activation barriers were approximately the same within experimental uncertainties (SiC–polymer: 1.01 eV; Si–polymer: 0.96 eV; SiC–SiO₂: 0.96 eV; DLC–SiO₂: 1.0 eV). In contrast, V_{eff} was much lower for the SiC tips (SiC–polymer: 2.0×10^{-29} m³, SiC–SiO₂: 2×10^{-29} m³, Si–polymer: 3.4×10^{-28} m³, DLC–SiO₂: 3.4×10^{-28} m³), a result that was also indicated by the more linear wear progression of the SiC tips (Figure 4 and 5). Therefore, the wear improvement is expected to be even greater for sharper tips and more moderate for blunt tips. Both these limits are beyond the regime experimentally addressed here (see Supporting Information).

Whether these differences originate from differences in the frictional properties (ζ) or from the bond-breaking process (V_{act}) is a question for future study. To understand the molecular origins of wear of SiC, and to test the applicability of this or alternative wear models further, data on the pressure and temperature dependence of the friction are required.

5. Conclusions

In summary, we have developed a novel process for terminating Si tips with an ultrathin SiC layer that preserves the nanoscale sharpness of the tip and endows it with several orders of magnitude improvement in wear resistance. The wear behavior is well-fitted by an atomic bond-breaking model, which enables prediction of the durability of the tip. The sharpness, high wear resistance and high thermal stability of these tips make them well-suited to thermomechanical data storage and thermomechanical lithography. This transformative development may enable more widespread technological use of thermomechanical probe techniques and scanning probe applications in general. Moreover, the chemical inertness of SiC, already shown to be beneficial for micro-electromechanical systems (MEMS) applications at elevated temperatures,^[32,33] may lead to other applications in harsh chemical environments as well as in nano-electromechanical systems (NEMS).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Acknowledgements

The authors gratefully acknowledge Ute Drechsler and Michel Despont for the cantilever fabrication, Richard Stutz for the RTA processing, Ingmar Meijer for support using the FTIR spectroscopy, Rachel Cannara for help during the TEM characterization, Tobias Kraus and Matthew Hammilton for the XPS measurements, and the IBM probe storage teams for support and discussions. R.W.C. and K.S. gratefully acknowledge financial support from IBM Research - Zürich. R.W.C. gratefully acknowledges financial support from the National Science Foundation under grant CMMI-0826076. This research was partially supported by the Nano/Bio Interface Center through the National Science Foundation NSEC DMR08-32802.

> Received: October 4, 2011 Published online: February 8, 2012

- [1] X. N. Xie, H. J. Chung, C. H. Sow, A. T. S. Wee, Mater. Sci. Eng. Rep. 2006, 54, 1–48.
- [2] D. Pires, J. L. Hedrick, A. De Silva, J. Frommer, B. Gotsmann, H. Wolf, M. Despont, U. Duerig, A. W. Knoll, *Science* 2010, 328, 732–735.
- [3] A. W. Knoll, D. Pires, O. Coulembier, P. Dubois, J. L. Hedrick, J. Frommer, U. Duerig, Adv. Mater. 2010, 22, 3361–3365.
- [4] O. Coulembier, A. Knoll, D. Pires, B. Gotsmann, U. Duerig, J. Frommer, R. D. Miller, P. Dubois, J. L. Hedrick, *Macromolecules* 2010, 43, 572–574.
- [5] R. D. Piner, Z. Jin, X. Feng, H. Seunghun, C. Mirkin, Science 1999, 283, 661–663.
- [6] C. S. Mirkin, ACS Nano 2007, 1, 79-83.
- [7] S. Xu, N. A. Amro, G. Y. Liu, Appl. Surf. Sci. 2001, 175–176, 649–655.
- [8] S. Cruchon-Dupeyrat, S. Porthun, G. Y. Liu, Appl. Surf. Sci. 2001, 175–176, 636–642.
- [9] M. R. Falvo, G. Clary, A. Helser, S. Paulson, R. M. Taylor, V. Chi, F. P. Brooks, S. Washburn, R. Superfine, *Microsc. Microanal.* **1998**, 4, 504–512.
- [10] T. R. Ramachandran, C. Baur, A. Bugacov, A. Madhukar, B. E. Koel, A. Requicha, C. Gazen, *Nanotechnology* **1998**, *9*, 237–245.
- [11] M. Sitti, H. Hashimoto, in Proc. 1998 IEEE/RSJ Int. Conf. Intelligent Robots Systems., Volume 3, IEEE, Piscataway, NJ 1998, 1739–1746.
- [12] M. F. Crommie, C. P. Lutz, D. M. Eigler, Science 1993, 262, 218–220.
- [13] C. M. Lieber, Y. Kim, Adv. Mater. 1993, 5, 392-394.
- P. Vettiger, G. Cross, M. Despont, U. Drechsler, U. Durig, B. Gotsmann, W. Haberle, M. A. Lantz, H. E. Rothuizen, R. Stutz, G. K. Binnig, *IEEE Trans. Nanotechnol.* 2002, 1, 39–55.
- [15] E. Eleftheriou, T. Antonakopoulos, G. K. Binnig, G. Cherubini, M. Despont, A. Dholakia, U. Durig, M. A. Lantz, H. Pozidis, H. E. Rothuizen, P. Vettiger, *IEEE Trans. Magn.* 2003, *39*, 938–945.
- [16] A. Pantazi, A. Sebastian, T. A. Antonakopoulos, P. Bächtold, A. R. Bonaccio, J. Bonan, G. Cherubini, M. Despont, R. A. DiPietro, U. Drechsler, U. Dürig, B. Gotsmann, W. Häberle, C. Hagleitner, J. Hedrick, D. Jubin, A. Knoll, M. A. Lantz, J. Pentarakis, H. Pozidis, R. C. Pratt, H. Rothuizen, R. Stutz, M. Varsamou, D. Wiesmann, E. Eleftheriou, *IBM J. Res. Dev.* **2008**, *52*, 493–511.
- [17] D. Wiesmann, C. Rawlings, R. Vecchione, F. Porro, B. Gotsmann, A. Knoll, D. Pires, U. Duerig, *Nano Lett.* 2009, *9*, 3171–3176.





www.afm-journal.de

- [18] R. J. Cannara, B. Gotsmann, A. Knoll, U. Dürig, Nanotechnology 2008, 19, 395305.
- [19] M. Hinz, O. Marti, B. Gotsmann, M. A. Lantz, U. T. Duerig, Appl. Phys. Lett. 2008, 92, 043122.
- [20] B. Gotsmann, M. A. Lantz, Phys. Rev. Lett. 2008, 101, 125501.
- [21] J. Liu, D. S. Grierson, N. Moldovan, J. Notbohm, S. Li, P. Jaroenapibal, S. D. O'Connor, A. V. Sumant, N. Neelakantan, J. A. Carlisle, K. T. Turner, R. W. Carpick, *Small* **2010**, *6*, 1140–1149.
- [22] Ph. Niedermann, W. Hanni, N. Blanc, R. Christoph, J. Burger, J. Vac. Sci. Technol. A 1996, 14, 1233–1236.
- [23] Ph. Niedermann, W. Hänni, D. Morel, A. Perret1, N. Skinner, P. F. Indermühle, N. F. de Rooij, P. A. Buffat, *Appl. Phys. A – Mater. Sci. Process.* **1998**, *66*, S31–S34.
- [24] R. P. Ried, H. J. Mamin, D. J. Rugar, *Microelectromech. Syst.* 2000, 9, 52–57.
- [25] K.-H. Kim, N. Moldovan, C. Ke, H. D. Espinosa, X. Xiao, J. A. Carlisle, O. Auciello, Small 2005, 1, 866–874.
- [26] H. Bhaskaran, B. Gotsmann, A. Sebastian, U. Drechsler, M. A. Lantz, M. Despont1, P. Jaroenapibal, R. W. Carpick, Y. Chen, K. Sridharan, *Nat. Nanotechnol.* 2010, *5*, 181–185.
- [27] D. R. Tallant, J. E. Parmeter, M. P. Siegal, R. L. Simpson, *Diamond Relat. Mater.* **1995**, *4*, 191–199.
- [28] R. I. Scace, G. A. Slack, in Silicon Carbide– A High Temperature Semiconductor: Proceedings of the Conference on Silicon Carbide, Boston, Massachusetts, April 2-3 1959, (Eds: J. R. O'Connor, J. Smittens) Pergamon Press, Oxford, **1960**, p. 24.
- [29] Y. Goldberg, M. E. Levinshtein, S. L. Rumyantsev, in Properties of Advanced Semiconductor Materials GaN, AlN, SiC, BN, SiC, SiGe, (Eds: M. E. Levinshtein, S. L. Rumyantsev, M. S. Shur), John Wiley & Sons, Inc., New York 2001, Ch. 5.
- [30] C. J. Glassbrenner, G. A. Slack, Phys. Rev. 1964, 134, A1058-A1069.
- [31] F. Liao, S. L. Girshick, W. M. Mook, W. W. Gerberich, M. R. Zachariah, *Appl. Phys. Lett.* 2005, *86*, 171913.
- [32] E. Hailu, A. R. Atwell, J. S. Duster, C. Li, M. Balseanu, K. T. Kornegay, in Nanotech 2003 Vol. 1, Technical Proceedings of the 2003 Nanotechnology

Conference and Trade Show, Volume 1, Nano Science and Technology Institute, Cambridge, MA **2003**, Ch. 10, p. 270–272.

- [33] T. -H. Lee, K. M. Speer, X. A. Fu, S. Bhunia, M. Mehregany, in Proc. Int. Solid-State Sensors, Actuators and Microsystems Conf., TRANS-DUCERS 2009, IEEE, Piscataway, NJ 2009, p. 900–903.
- [34] M. Despont, J. Brugger, U. Drechsler, U. Durig, W. Haberle, M. Lutwyche, H. Rothuizen, R. Stutz, R. Widmer, G. Binnig, H. Rohrer, P. Vettiger, *Sens. Actuators A* 2000, *80*, 100–107.
- [35] Handbook of Plasma Immersion Ion Implantation and Deposition, (Ed: A. Anders), Wiley Interscience, New York 2000.
- [36] R. R. Reeber, K. Sridharan, Adv. Mater. Processes 1994, 146, 21–23.
- [37] D. A. Glocker, Handbook of Thin Film Process Technology, (Eds: D. A. Glocker, J. I. Shah), Institute of Physics Publishing, Bristol 1995.
- [38] Z. An, R. K. Y. Fu, P. Chen, W. Liu, P. K. Chu, C. Lin, J. Vac. Sci. Technol. B 2003, 21, 1375–1379.
- [39] H. Miura, K. Yasui, K. Abe, A. Masuda, Y. Kuroki, H. Nishiyama, M. Takata, Y. Inoue, T. Akahane, Jpn. J. Appl. Phys. 2008, 47, 569–572.
- [40] A. Tachibana, Y. Kurosaki, K. Yamaguchi, T. Yamabet, J. Phys. Chem. 1991, 95, 6849–6854.
- [41] W. Wu, D. H. Chen, W. Y. Cheung, J. B. Xu, S. P. Wong, R. W. M. Kwok, I. H. Wilson, *Appl. Phys. A* **1998**, *66*, 5539–5543.
- [42] L. Moro, A. Paul, D. C. Lorents, R. Malhotra, R. S. Ruoff, P. Lazzeri, L. Vanzetti, A. Lui, S. Subramoney, J. Appl. Phys. 1997, 81, 6141–6146.
- [43] C. Pantea, G. A. Voronin, T. W. Zerda, J. Zhang, L. Wang, Y. Wang, T. Uchida, Y. Zhao, *Diamond Relat. Mater.* 2005, 14, 1611–1615.
- [44] A. Avila, I. Monter, L. Galán, J. M. Ripalda, R. Levy, J. Appl. Phys. 2001, 89, 212–216.
- [45] A. R. Chourasia, Surf. Sci. Spectra 2001, 8, 45-55.
- [46] C. Önneby, C. G. Pantano, J. Vac. Sci. Technol. A 1997, 15, 1597–1602.
- [47] B. Gotsmann, A. W. Knoll, R. Pratt, J. Frommer, J. L. Hedrick, U. Duerig, Adv. Funct. Mater. 2010, 20, 1276–1284.
- [48] T. D. B. Jacobs, B. Gotsmann, M. A. Lantz, R. W. Carpick, *Tribol. Lett.* 2010, 39, 257–271.