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Atom Scale Characterization of the Near Apex Region of an Atomic Force Microscope Tip

Christopher J. Tourek and Sriram Sundararajan*

Abstract: Three-dimensional atom probe tomography (APT) is successfully used to analyze the near-apex regions of an atomic force microscope (AFM) tip. Atom scale material structure and chemistry from APT analysis for standard silicon AFM tips and silicon AFM tips coated with a thin film of Cu is presented. Comparison of the thin film data with that observed using transmission electron microscopy indicates that APT can be reliably used to investigate the material structure and chemistry of the apex of an AFM tip at near atomic scales.

Key words: atomic force microscope (AFM), atom probe microscope, 3D atom probe tomography (APM), thin film

INTRODUCTION

Since its invention in the early 1980s, atomic force microscopy (AFM) or scanning probe microscopy (SPM) has become one of the most powerful tools in the fields of nanoscience and nanotechnology for the preparation and analysis of materials, nanostructures, and their functionality (Bhushan et al., 1995; Carpick & Salmeron, 1997; Hansma & Pietrasanta, 1998; Lillehei & Bottomley, 2000; Oesterschulze, 2001; Greene et al., 2004; Poggi et al., 2004; Loos, 2005). Over the years, the basic principle of SPM—measuring a specific interaction between an ultra-sharp tip and a material’s surface to collect structural or functional information—has generated a complete family of techniques such as electrical force microscopy (Gewirth & Niece, 1997; Kronik & Shapira, 1999; Berger et al., 2009), magnetic force microscopy (Hartmann, 1999; Gruverman & Kholkin, 2006; Kalinin et al., 2007), and scanning probe lithography (Holmberg et al., 2000; Wouters & Schubert, 2004; Xie et al., 2006) to name a few. Using specific tips and measuring conditions, a whole range of data can be measured (Magonov & Reneker, 1997; Giancarlo & Flynn, 1998; Horber & Miles, 2003; Samori, 2004; Santos & Castanho, 2004; Butt et al., 2005; Nicholls et al., 2005; Muller & Dufrene, 2008). Analyzing the geometry of the near apex region of the AFM tip can provide useful information for various applications including investigation of modified probes (e.g., nanotube attachments) (Cheung et al., 2000; Wade et al., 2004; Martinez et al., 2005) or for in situ analysis of tip shape during experiments (Fujisawa & Kizuka, 2003). Detailed assessment of the structure and chemistry of the near apex region of the AFM tip can greatly expand the analysis capabilities afforded by AFM. Specific areas include fabrication and wear using AFM tips where material transfer and tip chemistry are of importance (Chung & Kim, 2007; Karuppiah et al., 2009a, 2009b; Tanaka et al., 2009). To our knowledge, such analysis of AFM tips has not been reported in the literature.

This article presents the results of a study aimed at using three-dimensional (3D) atom probe tomography (APT) to analyze the structure and chemistry of the near-apex regions of uncoated AFM tips as well as tips coated with a thin film of known composition. APT is a technique capable of concurrently determining 3D material structure and chemistry at near atomic resolution, which has seen expanding use in science and engineering fields (Larson et al., 1999; Danoix & Auger, 2000; Hono & Ping, 2000; Hono, 2002; Gopalan et al., 2004; Gorman et al., 2007; Thompson et al., 2006; Kelly et al., 2007; Kelly & Miller, 2007; Seidman, 2007; Perea et al., 2008; Cojocaru-Mirendin et al., 2009).

ATOM PROBE TOMOGRAPHY

APT works on the phenomena of field evaporation, by which surface atoms of the specimen are ionized in ultrahigh vacuum and subsequently desorbed by an electric field (Muller & Bahadur, 1956). A schematic of the APT instrumentation is shown in Figure 1. The field evaporation
A short voltage pulse set up between the sample and the local electrode is used to ionize and remove an atom from the surface of the sample. It then passes through a hole in the local electrode toward a wide-angle position-sensitive detector equipped with a time-of-flight mass spectrometer with single atom sensitivity. The pulsed voltage on the local electrode is used to increase the electric field magnitude for evaporation, and the standing voltage on the sample is kept close to the voltage needed for field evaporation. The ionized atom is then repelled through a hole in the local electrode toward a wide-angle position-sensitive detector equipped with a time-of-flight mass spectrometer with single atom sensitivity. Typical magnifications of the specimen at the detector in a pulsed voltage mode at 200 kHz, using a 15% pulse fraction and a 160 mm flight path. The voltage was in-

**Material and Methods**

AFM tips are typically conical or pyramidal and have an apex radius ranging from tens of nanometers to microns depending on the type of investigation and the dimensions of the feature of interest. The similar tip geometry of AFM tips compared to a traditionally fabricated APT sample makes it a good candidate for APT.

In this study, two different commercially available AFM tips were studied—NSC15 (MikroMasch, Portland, OR, USA) and HAR5 (Nanoscience Instruments, Inc., Phoenix, AZ, USA). While both the tips are made from silicon (Si) oriented along the (100) direction, the NSC15 has faceted sides and the HAR5 is smooth as shown in Figure 2. The HAR5 also has a larger aspect ratio (5:1 compared to 3:1 for NSC15), and the backside of the cantilever is coated with aluminum for reflectivity in AFM experiments. For comparison purposes, standard Si microtip specimens (fabricated array of sharp Si tips on a Si substrate available from IMAGO Scientific Instruments, Madison, WI, USA) were also studied. The microtips provide consistent data and have been well studied (Thompson et al., 2007). Both the AFM tips and the microtip specimens were mounted using a commercially available clip holder (IMAGO Scientific Instruments) to allow analysis of the AFM tips without the need for specialized or permanent mounting.

In addition to uncoated tips, a NSC15 AFM tip and a microtip coupon were sputter deposited with a simple thin film with known composition to determine the ability to analyze a thin film on an AFM tip. Cu was chosen for the film primarily because of its similar evaporative field compared to Si. This reduces the stress when field evaporating through the interface. The tips were plasma cleaned for 30 s before depositing Cu for 2 min at 0.11 nm/s to give an estimated film thickness of 13 nm.

The samples were analyzed using a LEAP 3000x (IMAGO) atom probe microscope in voltage mode. Experiments were conducted at $4 \times 10^{-11}$ Torr or less, and the samples were cooled to at least 75 K. All data were collected in a pulsed voltage mode at 200 kHz, using a 15% pulse fraction and a 160 mm flight path. The voltage was in-

**Figure 1.** Diagram of an atom probe microscope. A short voltage pulse set up between the sample and the local electrode is used to ionize and remove an atom from the surface of the sample. It then passes through a hole in the local electrode to hit a position sensitive detector.
creased slowly until the tip started to field evaporate, and at least 900,000 atoms were collected in each experiment. After data collection the atom positions from the runs were reconstructed utilizing IVAS analysis software using the known tip radius of the sample and the evaporation field of Si. The approximate measured volume for the reconstructions presented here are 30 nm x 30 nm x 30 nm for uncoated samples and 30 nm x 30 nm x 80 nm for coated samples.

We note that radii of the AFM tips were obtained experimentally by reverse imaging a sharp tip characterizer sample (TGT-1, NT-MDT) in a Dimension 3100 AFM (Veeco Instruments, Santa Barbara, CA, USA) and reconstructing the tip shape using established methods (Williams et al., 1996; Villarrubia, 1997; Bykov et al., 1998). The tip radius is one of the parameters used in the software-based reconstruction of the atom positions in APT. Consequently the ability to determine a priori a relatively accurate tip radius helps minimize errors in the reconstruction (Miller, 2000). The tip radii for the AFM tips in this study were on the order of tens of nanometers. Figure 3 shows the tip profile of the HAR 5 tip that was used to obtain the data shown in Figure 4b.

The first 10 nanometers of the reconstructions of uncoated AFM tips and microtip are shown in Figure 4. The data show dark regions caused by local trajectory aberrations in the field evaporation of the Si atoms around the ^{100} pole, confirming that both AFM tip types and the microtips are composed of Si orientated along the ^{100} direction (Vurpillot et al., 1999). The data lend confidence to the feasibility of analyzing AFM tips using APT.

A transmission electron microscopy (TEM) analysis of the Cu coated NSC15 tip yielded a film thickness estimate of 11 nm as shown in Figure 5. Figures 6 and 7 show a slice through the APT data for a coated NSC15 tip and microtip, respectively. The slices display only a percentage of the atoms for visual clarity. The Cu layer and the Si bulk can be easily discerned, and the inset in each figure shows a magnified view that highlights the interface. The estimated film thickness from the APT data on the NSC15 and the microtip were 11 nm and 10 nm, respectively, which agree very well with the expected film thickness from the TEM observation.

We note that the AFM cantilevers are free to bend when mounted on the clip holder in the atom probe microscope, and the electric field present in APT creates a force on the tip being analyzed (Mayama et al., 2007). This force causes the AFM cantilever to bend during analysis, which can affect the reconstruction because the tip axis will no longer be perfectly aligned with the local electrode axis during the field evaporative process. In preliminary tests with cantilevers of spring constants from 0.3 to 2 N/m, it was seen that the bending could cause the tip to contact the local electrode, and hence we moved to much stiffer cantilevers. Simple finite element modeling in which a constant electro-
static field was assumed suggested that for the cantilevers used here [calibrated (Sader et al., 1999) normal stiffness of 25 N/m], the cantilever bending resulted in less than 5° total axis misalignment between the tip and the local electrode over the total voltage range used for the experiments. A propagation of error calculation in the reconstructed position of an atom caused by a sample tilt of this order resulted in an upper bound error estimate of 9 Å for the lateral dimension and 2 Å for the depth coordinate. We utilized the reconstruction method outlined by Bas et al. (1995) for our calculations. It is noted that this estimate is for a static deflection (for a given electric field), and a more detailed analysis on its effect on the APT data, especially in the real case of increasing field strength as the experiment progresses, is warranted. Continuous cantilever bending during the experiment can therefore contribute to considerable error in atom position data. In our study, we did not notice

Figure 4. The first 10 nm of the (a) NSC15, (b) HAR5, and (c) microtip reconstructions looking down the analysis direction. All dimensions are in nm. The Si (100) pole is highlighted.

Figure 5. TEM image of a NSC15 AFM tip coated with Cu. The film thickness is highlighted.

Figure 6. A slice of the reconstruction of a NSC15 tip sputter deposited with Cu with a blowup of the center of the analysis to show the Cu film thickness. Dimensions are in nm.
continuous bending once the electric field reached the field evaporative point. In addition, with the film thicknesses of 11 nm as verified by TEM, the error in the depth coordinate of 2 Å did not significantly affect the values of film thickness obtained from APT analysis. It is worth noting that if the APT analysis is only concerned with material chemistry such as percent solute analysis, then the spatial error is not of great concern, especially if enough data are collected for proper statistics (Miller et al., 1996).

**CONCLUSIONS**

APT analysis was completed on both uncoated and thin film coated AFM tips, and the results indicate that APT is a viable technique to obtain near atom-scale information of material structure and chemistry of the near-apex regions of such tips. This opens up a new avenue of investigation for AFM-related research. Possible topics of investigation range from assessing the durability and verifying the structure of thin films and coatings on an AFM tip to characterizing the structure and chemistry of transferred or newly generated material on the tip. Critical factors in the experiments were stiffness of the cantilever and mounting of the AFM tip in the atom probe microscope. Cantilevers with normal stiffness of 25 N/m and above appear to be viable for APT analysis. Our current and future work is aimed at a more detailed assessment of the effect of cantilever bending on the reconstruction and the analysis of transfer films from AFM-based wear studies.

**ACKNOWLEDGMENTS**

Funding for this study was provided by a grant from the National Science Foundation (Grant No. 0932573). The authors would also like to thank Matthew Kramer, from Ames Laboratory, for assistance in TEM analysis.

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