Title
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Journal
Journal of Vacuum Science & Technology B Nanotechnology and Microelectronics Materials Processing Measurement and Phenomena, 35(5)

ISSN
2166-2746

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Publication Date
2017-09-01

DOI
10.1116/1.4994315

Peer reviewed

SHOP NOTES

These are "how to do it" papers. They should be written and illustrated so that the reader may easily follow whatever instruction or advice is being given.

Minimizing open-loop piezoactuator nonlinearity artifacts in atomic force microscope measurements

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(Received 5 July 2017; accepted 25 August 2017; published 8 September 2017)

Atomic force microscopes (AFMs) are widely used to study molecular interactions with piconewton force sensitivity. In an AFM, interaction forces are measured by reflecting a laser beam off a cantilever onto a position sensitive detector and monitoring cantilever deflection. Precise measurements of interaction forces rely on accurately determining the optical lever sensitivity, i.e., the relationship between cantilever deflection and changes in detector voltage. The optical lever sensitivity is measured by pressing the cantilever against a hard substrate using a piezoactuator and recording the resulting change in detector voltage. However, nonlinearities in the motion of commonly used open-loop piezo actuators introduce significant errors in measured optical lever sensitivities. Here, the authors systematically characterize the effect of piezo actuator hysteresis and creep on errors in optical lever sensitivity and identify measurement conditions that minimize these errors. © *2017 American Vacuum Society*. [http://dx.doi.org/10.1116/1.4994315]

I. INTRODUCTION

Atomic force microscopes (AFMs) are widely used to measure structural properties and inter/intramolecular forces with single molecule sensitivity.[1–3] In a typical AFM experiment, forces are measured by monitoring the deflection of a micrometer-sized cantilever with a sharp tip at one end. To measure cantilever deflection, AFMs commonly utilize an optical lever setup where a laser beam is reflected off the back side of the cantilever onto a position sensitive quadrant photodiode (QPD). As the cantilever bends, the position of the laser beam on the QPD changes, which results in a change in difference voltage (ΔV). The deflection of the cantilever is determined by measuring the inverse optical lever sensitivity (sensitivity, S), which is the relationship between cantilever deflection (ΔD) and ΔV.[4,5] Accurate values of measured sensitivity are critical for determining interaction forces, calculating cantilever spring constants, and calculating the distance between the AFM tip and the underlying substrate.[6,7]

Sensitivity is typically measured by pressing the cantilever on a hard, nondeformable surface and then moving it by a known distance, ΔD, relative to an arbitrary reference point, while simultaneously monitoring the voltage change in the QPD. The sensitivity is calculated as: $S = \Delta D/\Delta V$. In these measurements, either the cantilever or the sample is translated using open-loop or closed-loop piezoelectric actuators (PZTs). While closed-loop PZT displacement is characterized by its linearity, repeatability, and accuracy, the displacement of an open-loop PZT is nonlinear due to hysteresis and creep.[8–10] These nonlinearities in open-loop PZT motion can contribute to substantial errors in ΔD, which results in sensitivity errors.[11,12] While methods have been developed for correcting sensitivity errors by calibrating the response of QPDs,[13] quantitative measurements of sensitivity errors due to PZT nonlinearity and simple strategies to minimize these errors have not been explored.

In this study, we characterize the effect of the open-loop PZT travel range, travel direction, travel rate, and dwell times on ΔD and sensitivity errors. We show that these errors are dramatically reduced when an open-loop PZT is operated around the region where its travel is most linear. This linear travel region can be easily measured using established methods for measuring hysteresis in open-loop PZT motion.[14,15]

II. RESULTS

We systematically measured the motion of closed-loop and open-loop PZTs, identified regions with linear travel, and compared optical lever sensitivities in the linear and nonlinear travel ranges. All measurements were performed using two PZT scanners (navy VI type piezoelectric ceramic, PZT5H2, Keysight Technologies), which could be operated in either closed-loop or open-loop modes and had travel ranges of 13.2 μm (+6.6 to −6.6 μm) and 16.6 μm (+8.3 to −8.3 μm), respectively. Built-in inductive position sensors allowed us to monitor the movement of the PZT at a data acquisition rate of 25 kHz. Linear travel was defined as the region of expected

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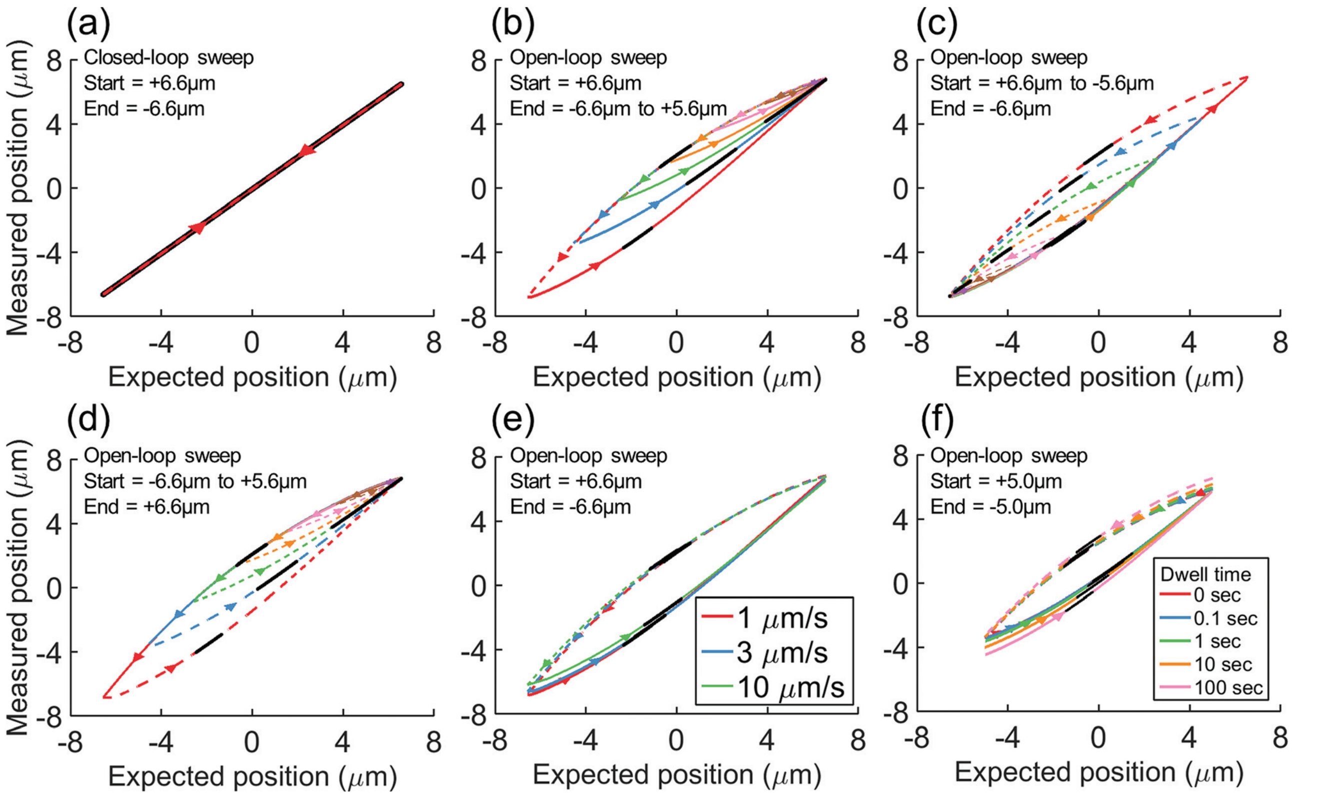

FIG. 1. (Color) Characterizing closed-loop and open-loop PZT linearity. For each measurement condition, ten consecutive sweeps were performed at a speed of 1 μm/s [except in (e)] and then averaged. Linear regions of travel are colored black. (a) Closed-loop PZT motion across the entire travel range was linear. PZT was swept between +6.6 and −6.6 μm (approach: dashed; withdrawal: solid). (b) and (c) The overlap and linearity of open-loop sweeps, depended on the sweep starting position. In (b), the PZT was moved from +6.6 μm to different end positions (dashed lines) and then back to +6.6 μm (solid lines). In (c), the PZT was moved from different start positions to −6.6 μm (dashed lines) and back to the start positions (solid lines). (d) Sweep direction did not affect open-loop linearity. PZT was moved from different start positions to +6.6 μm (dashed lines) and back to the start positions (solid lines). (e) Effect of the sweep rate on open-loop linearity. PZT was moved from +6.6 to −6.6 μm (dashed lines) and back to +6.6 μm (solid lines) at different sweep rates. (f) Effect of the open-loop PZT creep. Different dwell-times were introduced between sweeps in the approach (dashed lines) and retraction (solid lines) directions as the PZT was swept between +5.0 and −5.0 μm.

versus measured PZT position plots that had a slope that was within the range of 1 ± 0.05.

First, we characterized the linearity of the closed-loop and open-loop PZT by sweeping it across its complete travel range [Figs. 1(a) and 1(b)]. The PZT was swept from the positive to negative extremum (approach sweep) and then swept back to the positive extremum (withdrawal sweep) at a speed of 1 μm/s. As expected, closed-loop PZT motion across the entire travel range was linear with a slope of 1.0001 ± 0.0002 and 1.0000 ± 0.0001 for the approach and withdrawal sweeps, respectively. In contrast, when the PZT was operated in the open-loop mode, only 9.1% of the entire approach movement (+0.6 to −0.6 μm) and 9.5% of the entire withdrawal sweep (−2.4 to −1.1 μm) were linear [Figs. 1(b) and 1(c)]. Similarly, we measured a linear travel range of 9.6% (+0.7 to −0.9 μm) in the approach sweep and 10.9% (−2.4 to −0.6 μm) in the withdrawal sweep, using a second open-loop PZT with a total travel of 16.6 μm (supplementary material Fig. 1).[19]

Next, we characterized the effect of the open-loop PZT travel range on nonlinearity and consequently on optical lever sensitivity. During the approach sweep, the PZT was translated from the same starting position (+6.6 μm) to different ending positions (−6.6, −4.6, −2.6, −0.6, +1.6, +3.6, and +5.6 μm, respectively) at a speed of 1 μm/s. During the withdrawal sweep, the PZT returned to +6.6 μm at the same speed [Fig. 1(b)]. Thus, while all the approach sweeps started at the same position, the starting positions for each withdrawal sweep were different. Our measurements showed that the approach sweeps overlapped in all cases and linear travel was centered between −0.7 and +0.6 μm. In contrast, the withdrawal sweeps did not overlap and the linear travel did not occur within the same region of each sweep [Fig. 1(b)].

Since the linear region in Fig. 1(b) overlapped only during the approach sweep, we hypothesized that its location was determined by the starting position of the sweep. To test this hypothesis, we moved the PZT across a similar range and at the same speed as in Fig. 1(b) but changed the starting position of the approach sweep. Consequently, while we started the approach sweep at +6.6, +4.6, +2.6, +0.6, −1.6, −3.6, and −5.6 μm, respectively, every withdrawal sweep began at its negative extremum (−6.6 μm) [Fig. 1(c)]. As anticipated, the approach sweeps did not overlap and their linear regions were no longer centered on the same portion of the sweep. In contrast, the withdrawal sweeps, which now began at the same starting point, overlapped and the corresponding linear regions were all centered around the same portion of the sweep (−2.4 to −0.6 μm) [Fig. 1(c)].

To confirm that the position of the linear region in an open-loop sweep was dominated only by the starting position, we reversed the direction of the sweep [Fig. 1(d)]. We set the PZT to move with the same travel range and the same speed as in Fig. 1(b) but in an opposite direction by first executing a withdrawal sweep and then performing an approach sweep. The measured trajectories were identical to those shown in Fig. 1(b), indicating that the sweep sequence did not matter. Furthermore, while the linear region in the withdrawal traces did not overlap, the linear portion of the approach sweep, which all started at +6.6 μm, was centered between +0.6 and −0.6 μm [Fig. 1(d)].

Next, we characterized the effect of the sweep rate on linear movement; the open-loop PZT was swept between +6.6 and −6.6 μm at 1, 3, and 10 μm/s [Fig. 1(e)]. While the fraction of linear travel in approach sweeps was independent of PZT speed (linear travel of 9.1%, 8.8%, and 9.2% at 1, 3, and 10 μm/s, respectively), the fraction of linear travel in withdrawal sweeps slightly increased with the sweep rate (linear travel of 9.5%, 10.4%, and 12.5% at sweep rate of 1, 3, and 10 μm/s, respectively).

Finally, we characterized the nonlinearity of open-loop PZT travel at different dwell times, i.e., the time period the PZT is held at a constant voltage between approach or withdrawal sweeps. Dwell time is an important parameter in the operation of an open-loop PZT actuator since it is related to PZT creep under a constant drive voltage.[16] The PZT was swept between +5.0 and −5.0 μm at a speed of 1 μm/s; dwell times of 0, 0.1, 1, 10, and 100 s were used [Fig. 1(f)]. As the dwell time increased, the linear range in approach sweeps increased slightly from 9.6% to 10.7%. In contrast, the linear travel of withdrawal sweeps dropped from 17.1% to 11.7% [Fig. 1(f)].

Having characterized closed-loop and open-loop PZT motion, we proceeded to test if open-loop sensitivities measured during linear PZT travel were similar to the real (closed-loop) sensitivity. Since the linear regions in open-loop approach sweeps in Fig. 1(b) were centered between +0.6 and −0.6 μm, we measured the closed-loop and open-loop sensitivities at three positions in the approach sweep: +4 μm (nonlinear open-loop), 0 μm (linear open-loop), and −4 μm (nonlinear open-loop). A glass substrate was positioned such that a PZT mounted AFM tip came into contact with the substrate at a PZT position of +4, 0, or −4 μm. The PZT was then translated in the approaching direction until the increase in QPD voltage due to cantilever deflection reached 4 V (Fig. 2). Sensitivity was calculated as the inverse of the linear slope when the cantilever was pressed against the substrate (Fig. 2). Only cantilever deflection at relative PZT position <-100 nm was used in sensitivity analysis (Fig. 2). As anticipated from the linearized travel of the closed-loop PZT [Fig. 1(a)], our measurements showed that the closed-loop sensitivity does not change with the PZT position [Fig. 2(a)]. Sensitivities of 309, 312, and 314 nm/V were obtained at +4, 0, and −4 μm. In contrast, while open-loop sensitivity measured within the linear travel region (S = 283 nm/V at 0 μm) was similar to the closed-loop sensitivity, the open-loop sensitivity measured outside the PZT linear range had an error of up to 36% (S = 377 nm/V at +4 μm and S = 200 nm/V at −4 μm) [Fig. 2(b)].

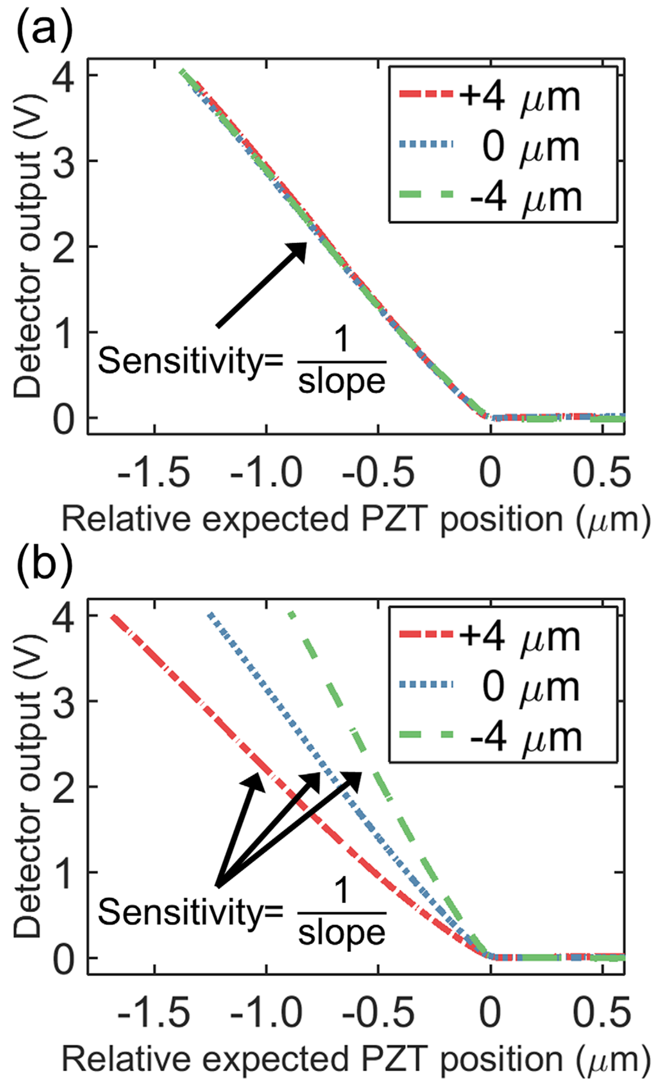

FIG. 2. (Color online) Optical lever sensitivity is accurate during linear PZT travel. Detector voltage vs PZT movement during (a) closed-loop and (b) open-loop operation. Sensitivity was measured at three different PZT positions. To enable direct comparison, the points of the tip-surface contact in all measurements were aligned to the origin.

We confirmed that we could always obtain accurate open-loop sensitivities during linear PZT travel, using eight different cantilevers (supplementary material, Table I). Sensitivities were measured, as shown in Fig. 2, and normalized errors were calculated as: $(S_{\text{open-loop}} - S_{\text{closed-loop}})/S_{\text{closed-loop}}$. Average errors of 29.7 ± 5.6%, −4.2 ± 2.7%, and −32.9 ± 1.9% were obtained when open-loop sensitivities were measured at +4, 0, and −4 μm, respectively (supplementary material, Table I). When the measured sensitivities were used to calculate cantilever spring constants using the thermal fluctuation method[17] (supplementary material, Table II), an error of −40% and +122% was measured using open-loop sensitivities at +4 and −4 μm. In contrast, when the open-loop sensitivity measured at 0 μm was used in the calculation, the error in the spring constant was reduced to +9%.

III. DISCUSSION AND CONCLUSIONS

Our study characterizes the effect of PZT nonlinearity on the optical lever sensitivity of AFM cantilevers and identifies measurement conditions which minimize these errors. By systematically studying the effect of the travel range, travel direction, travel rate, and dwell times on the open-loop PZT linearity, we identify conditions where linear PZT motion can be measured. We show that the linear portion of open-loop PZT motion is largely controlled by the starting position of the sweep. For the open-loop PZTs used in this study, motion was linear and sensitivity was accurately measured, around the center of PZT travel during a full-range approach sweep. Measurements in this linear region reduced errors in sensitivity to -4% and errors in the spring constant to $+9\%$. In contrast, when sensitivity was measured in regions where travel was nonlinear, errors in the measured sensitivities and spring constants were up to 36% and 122%, respectively.

It is important to note that since our scanner was equipped with position sensors, we directly identified linear travel from the expected versus measured PZT position plots. However, linear travel in open-loop PZTs that do not have positon sensors can be easily measured by using simple methods such as scanning a tilted smooth surface[14] or using a small mirror to perform laser interferometry.[15] Furthermore, while linear motion in our PZT was measured around the center of travel, this might hold only for PZTs that have a voltage drive which changes symmetrically between positive and negative values. It is therefore important to measure the linear region of travel for any open-loop PZT[14,15] before using it for sensitivity measurements.

While our results clearly demonstrate that accurate sensitivities are best measured using closed-loop PZTs, the guidelines provided in this study also enable the accurate measurement of sensitivity using open-loop PZTs. Currently, there are ongoing efforts to standardize AFM spring constant calibrations and improve its reproducibility and accuracy.[18] Our results add to these efforts and will facilitate the accurate measurement of interaction forces in AFM experiments.

ACKNOWLEDGMENT

This work was supported by the National Institute of General Medical Sciences of the National Institutes of Health under award No. R01GM121885.

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[19]See supplementary material at http://dx.doi.org/10.1116/1.4994315 for linearity characterization of an additional open-loop PZT, and for tabulation of errors in open-loop sensitivity measurements and spring constant calculations.