JOURNAL OF CHEMICAL EDUCATION

pubs.acs.org/jchemeduc Laboratory Experiment

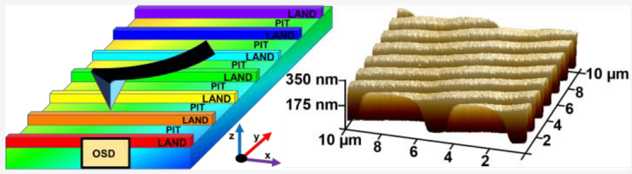
An Inquiry-Based Introduction to Atomic Force Microscopy Techniques through Optical Storage Disc Surface Imaging

Senetta F. Bancroft,* M. Aswad Ali, and Punit Kohli

Cite This: J. Chem. Educ. 2022, 99, 3030−3038 Read Online

ACCESS | Metrics & More | Article Recommendations | Supporting Information

ABSTRACT: Scanning probe microscopy such as atomic force microscopy has become increasingly integrated and relevant in undergraduate laboratory investigations. As the "hands and eyes of the nanoworld", atomic force microscopes (AFMs) continue to be used to innovate the nanowriting process for data storage and in the exploration of novel materials suitable for ultrafast, nonvolatile, high density memory storage. Optical storage discs (OSDs) coevolved with AFM technology, are relatively inexpensive, and are easily available for purchase. They can, consequently, serve as a relevant phenomenon for undergraduate students to explore nanoscale material structure and macroscopic function through an AFM. The guided inquiry investigation presented here introduces students to the design and basic functions of an AFM through their identification of three unknown OSD samples (CD, DVD, or Blu-ray). The surface of each OSD is explored through students' collection and interpretation of topographic and force curve data using an AFM to identify differences in the structure of the grooves containing pits and lands, estimation of theoretical storage capacity, and finally distinguish OSD types by comparing their data and calculations to known structures and capacities.



KEYWORDS: *Upper-Division Undergraduate, Analytical Chemistry, Inquiry, Materials Science, Nanotechnology, Surface Science*

■ INTRODUCTION

Data Storage as a Foundational Context for Learning AFM Techniques

The American Chemical Society (ACS) encourages undergraduate curricula to incorporate laboratory experiences that include the preparation, characterization, and physical properties of systems including nanoscale materials.[1] Scanning probe microscopy using instruments such as an atomic force microscope (AFM), subsequently, has become increasingly integrated and relevant in undergraduate laboratory investigations.[2−6] Optical storage discs (OSDs) coevolved with AFM technology and, thus, can serve as relevant phenomenon for students to explore through an AFM within an undergraduate curriculum.

In 1999, Binnig and colleagues introduced an AFM based technique for erasable high density data storage using thermomechanical writing.[7] Data storage occurs through indentations formed by a heated a cantilever tip on a thin film such as polycarbonate disk surface. Innovations have since allowed for nanowriting without the use of heat and other modified polymer surfaces.[8] AFM, as the "hands and eyes of the nanoworld",[9] continues to be used to innovate the nanowriting process for data storage[10] and in the exploration of novel materials suitable for ultrafast, nonvolatile, high density memory storage.[11] There is literature[12,13] characterizing CDs, DVDs, and Blu-ray discs. However, there is a paucity of instructional resources suitable for adoption in an undergraduate chemistry curriculum supporting students' ability to learn AFM techniques needed to characterize OSDs and link their key submicroscopic physical properties to how data are stored. We have created an inquiry-based activity suited to this purpose.

Inquiry as a Framework for Sensemaking Laboratory Investigations

The ACS[1] has also called for undergraduate curricula to be delivered using practices derived from "modern theories of learning and cognition in science" (p. 5). Inquiry-based laboratory experiences, using theories such as constructivism, capitalize on actively engaging students to integrate prior and newly accumulated knowledge to build more complex representations of scientific concepts.[14] We use inquiry to refer the practices used by scientists during the process of investigating phenomenon (e.g., asking questions, obtaining background information, designing and carrying out procedures, data analysis, forming explanations, and communication of findings).[15] Different levels of inquiry require different levels of student decision making around these practices during investigations ranging from a level of 0 (confirmation inquiry),

Received: April 1, 2022
Revised: June 29, 2022
Published: July 12, 2022

https://doi.org/10.1021/acs.jchemed.2c00291
J. Chem. Educ. 2022, 99, 3030−3038

Table 1. Summary of Student Investigation and Alignment to Learning Objectives

Investigation Feature	Rationale
Pre-Lab	
• Read AFM background literature	• Provide students with sufficient prior knowledge to orient them to recognize key components of all AFMs (Obj. 1)
• Draw representations of generic AFM components	
Lab Results: Part 1	
• Draw, name, label, and describe the function of key components of Bruker Dimension Icon V AFM	• Associate key components of generic AFM with the Bruker Dimension Icon V they will be using to obtain data (Obj. 1)
Lab Results: Part 2	
• Observe TA preparation of disc samples and cantilever	• Experience, firsthand, how structural data are acquired using Bruker Dimension Icon V AFM (Obj. 2)
• Observe cantilever under microscope	
• Use AFM to scan unknown disc samples to obtain force curves and topographical maps	
Lab Results: Part 3	
• Organize force curves obtained for each region of three-disc samples	• Scaffold identification of differences and similarities in structural data of unknown disc samples (Obj. 2)
Lab Results: Part 4	
• Obtain characteristics of cantilever and tip used to obtain data	• Scaffold sensemaking around how cantilever and tip characteristics are integral to interpreting obtained data (Obj. 2, 3)
• Describe why these characteristics are important to consider when using an AFM	
Lab Data Analysis/Discussion	
• Quantitatively interpret surface topography data	• Scaffold sensemaking to infer function (amount of data storage and how data are stored) from disc structure data (Obj. 2, 3)
• Use analyzed data to identify each disc type and justify identification	
Lab Limitations	
• Account for any discrepancies in data	• Use acquired knowledge to make and justify decisions about the limitations in accuracy and precision of Bruker Dimension Icon V AFM (Obj. 4)
• Describe and justify what would be done differently if investigation was to be repeated	
Extending Your Learning	
• Describe how nc-AFM allows inference of chemical structure of molecules	• Use acquired knowledge of AFM functionality to make and justify decisions about use of AFM acquisition structural data in a new context (Obj. 5)

where the instructor makes all practice decisions, to level 3 (authentic inquiry), where students make all key practice decisions from identifying research questions to forming explanations about chemical phenomena.[16]

We designed a level 1 or guided inquiry investigation, where the instructor provided students with a problem to be investigated (identify unknown data storage disc samples), background information (determine key components of an AFM and their function to be able to obtain structure data to distinguish samples), and the procedure they must execute to obtain data (how to perform topographical scans using an AFM). Students make the remaining decisions to complete the investigative process with prompts scaffolding their ability to identify each disc sample using obtained data as evidence to support their decisions/claims. A guided inquiry investigation was determined to be appropriate because students were expected to have minimal background knowledge of the components of an AFM and corresponding techniques for obtaining physical property data.

Sensemaking Process Embedded within Inquiry

We further conceptualize our inquiry-based investigation as an overlapping instructional and assessment event designed to engage students in sensemaking around chemical phenomena. As an instructional event, the investigation is designed to produce learning around solving a problem using AFM techniques,[17] a problem students ultimately should solve through a series of scaffolded sensemaking processes around phenomenon and experimental data. The assessment event occurs when students' responses on their lab report are evaluated and is embedded within the instructional event (the investigation itself).[17]

Student decision making is a core characteristic of inquiry activities where students' prior knowledge interacts with the decision-making process aiding knowledge construction.[18] High levels of prior knowledge support student ability to accurately identify important relationships during instructional and assessment events.[19] At its most fundamental, successful decision making is evident when students can explain or predict outcomes by accurately mapping and connecting similarities and differences across chemical concepts and contexts.[18,20] Evaluation of their responses during assessment events related to the investigation are, therefore, a key source of determining whether a student can make sense of a phenomenon. Successful decision making throughout the investigative process for which students are responsible, therefore, reflects their ability to engage in sensemaking processes needed to problem solve or form explanations. In chemistry, this requires students to make connections among the macroscopic, submicroscopic, and symbolic levels of a chemical phenomenon.[21] A well-designed inquiry-based chemical investigation at any inquiry level, therefore, ensures students have sufficient prior knowledge of the phenomenon under investigation and engages them to actively make sense of the phenomenon across all three levels through data interpretation and scientific principles.[22]

https://doi.org/10.1021/acs.jchemed.2c00291
J. Chem. Educ. 2022, 99, 3030−3038

Using optical disc data storage as a phenomenon, our guided inquiry-based activity is designed to provide students with sufficient prior knowledge of the key components of an AFM, to infer storage capacity of discs from topographical and force curve data obtained from an AFM, and to apply their acquired knowledge to a new context in which modification of the AFM cantilever tip is conceptually used to infer properties of small molecules. This project was approved by Southern Illinois University Carbondale Institutional Review Board (Protocol # 21147).

Student sensemaking was driven by the experiment's purpose to identify three unknown disc samples as a CD, DVD, or Blu-ray (macroscopic conceptualization) using output data (submicroscopic conceptualization) and their inferences about the relationship between submicron or nanoscale structure of each sample and data storage capability (optical phenomenon). More specifically, given three prepared unknown disc samples, background literature,[23,24] and a procedure, students were supported to meet the following learning objectives:

1. Identify crucial AFM components and their function.
2. Acquire and interpret AFM data about the surface topography of three optical disc storage devices to estimate the amount of information each disc theoretically holds.
3. Describe how and why the tip and cantilever are the "heart" of the AFM by investigating the surface topography of three different hard disc storage devices.
4. Identify potential limitations and pitfalls of an AFM.
5. Apply acquired knowledge of AFM function to non-contact AFM (nc-AFM) techniques.

Table 1 outlines each section of the investigation relative to these objectives.

METHODS

Context

The lab described was implemented in Fall 2021 in a 400-level instrumental analysis course for chemistry majors taught by P.K.; a 300-level quantitative analysis course and lab was a prerequisite. Earlier versions of the lab were implemented in Fall 2019 and Fall 2020 with a total of 10 students. Five students were enrolled in the Fall 2021 course. In general, 2−4 students performed the lab as a group. Each student wrote their pre- and postlab reports individually. They, however, collaborated with each other in the interpretation of their data. From prelab to lab report submission, the experiment was completed over a four-week time span. Students submitted the prelab prior to two 4 h data collection lab sessions. They completed their analysis and submitted their written lab report 1 week following data collection completion. Teaching assistant M.A.A. facilitated students' engagement in the accompanying lab section for the course.

To further support knowledge extension, the laboratory was also supplemented with 3−4 lectures providing more fundamental and recent advances in AFM. Lectures were delivered after the laboratory. They captured five significant recent developments involving molecule-functionalized tips for image acquisition at the "*true-atomic*" level spatial resolution. First, the strength of a single hydrogen bond on a sample surface was determined with a tip modified with a thiol functional group.[25] Second, imaging CaF_2 in picometer lateral and horizontal resolution used a tip modified with a single CO molecule by interpreting the electrostatic interaction between tip and the sample surface.[26] Third, rotation of F1-ATPase at nanoscale spatial resolution provided a clear picture of structural basis of the torque generation in these nanoscale molecular motors.[27] Fourth, chemical structure[28] and bond order discrimination of aromatic molecules[29] were obtained using a CO-modified tip. Similarly, the CO-functionalized tips were demonstrated to image hydrogen bonding networks, including bonding cites, orientation, and lengths in 8-hydroxyquinoline molecules assemblies on a Cu (111) substrate.[30] Finally, the information regarding an organic reaction mechanism was obtained by imaging covalent structures of the traditional Bergman cyclization reaction using CO-modified AFM tips.[31] This final study was used to demonstrate how organic chemistry principles can be elucidated through collaboration with experimental AFM imaging experts.

Data were collected to determine the activity's short-term impact on participating student knowledge of AFM design and function through their lab report responses. Short-term impact of the lab on students' perception of their research experience, science identity, and self-efficacy were obtained through a presurvey administered 1 week prior to data collection and a postsurvey administered after students ($N = 4$) completed data collection 3 weeks later. The survey, adopted from Robnett and colleagues,[32] can be found in the Supporting Information. The student handout was revised on the basis of these data and the reflection of P.K. and M.A.A. on the Fall 2021 lab version's effectiveness to support student learning of AFM techniques. The revised student handout is included in the Supporting Information.

Limitations

Adoption of this lab experiment is limited to courses in which an AFM is available. The experiment was designed to focus on AFM components and functions common to AFMs and support students' learning of common AFM techniques such as sample preparation, sample loading, aligning the laser on the cantilever, optimizing the photodiode signal, and data analysis using freely available software such as Gwyddion and ImageJ. The OSDs used are widely available and inexpensive. However, some procedure details described are specific to the Bruker Dimension Icon V AFM. Therefore, the procedure and student handout provided should be modified to the AFM instrument available within local contexts. For students and instructors who do not have access to an AFM instrument, we have provided force−distance curves in text format in the Supporting Information. AFM micrographs are also available in .spm format and can be requested from the authors. Students and instructors, therefore, will be able to use and manipulate the data in MS office/Origin (for text files) and freely available Gwyddion software[33] (for .spm).

Prelab

To support students meeting learning objectives 1 and 2, the prelab requires them to read background literature[23,24] on the design and function of key components of an AFM and how to interpret force curve data. After reading background literature, students respond to the following open-ended prompts: (1) What is an AFM? and (2) Using appropriate schematic diagrams, cartoons, and figures, describe how an AFM works.

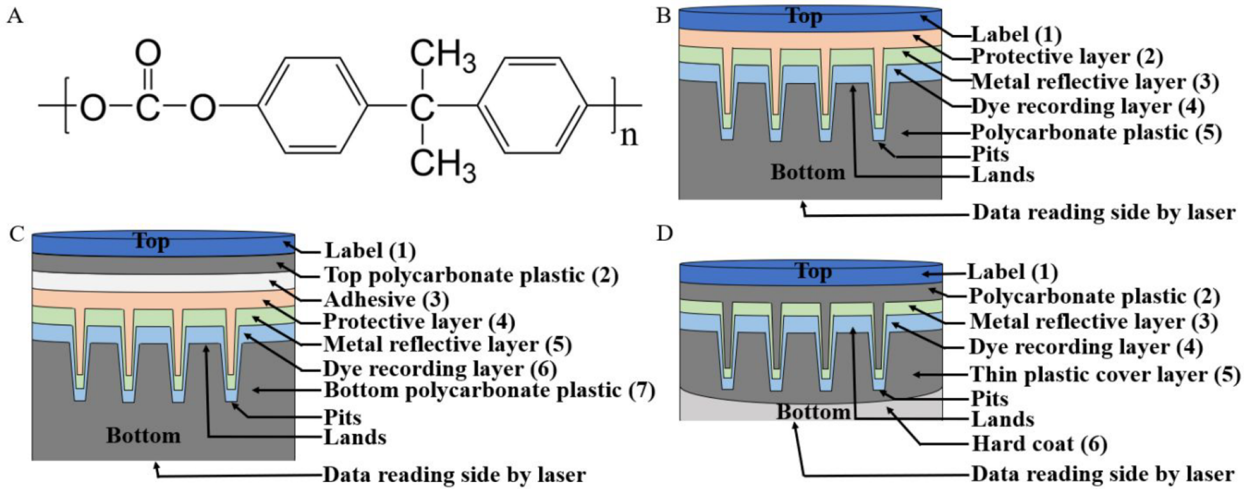


Figure 1. Anatomy of OSDs used in laboratory experiments. (A) Chemical structure of polycarbonate: a high performance polymer used to manufacture OSDs. Schematics of the architecture of a CD (B), DVD (C), and Blu-ray (D).

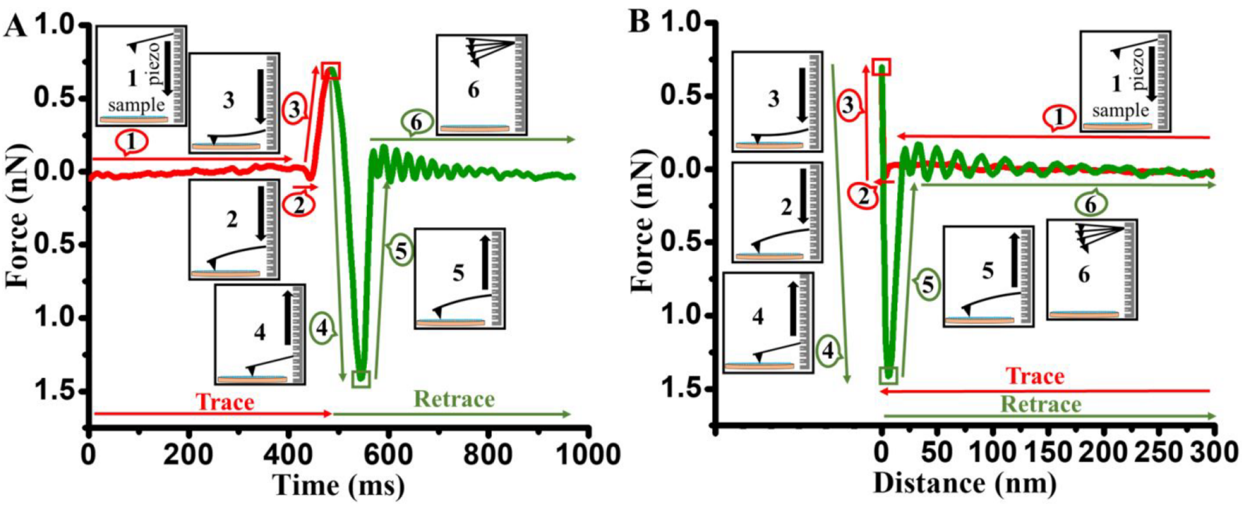


Figure 2. Illustration of force curve generation. (A) Trace and retrace scan lines in an average $F-t$ curve of Blu-ray. (B) Trace and retrace scan lines in an average $F-d$ curve of Blu-ray. Cantilever and piezo positions during a single scan on a sample are in the insets. Position 1, 2, 3, 4, 5, and 6 in the insets are directly correlated with six segments of a single trace−retrace line indicated by red and green bubbles and arrows.

Laboratory Investigation

Anatomy of OSDs. OSDs are generally composed of optically transparent polycarbonate. Figure 1A shows the chemical structure of polycarbonate. In general, all OSDs are multilayered where each layer serves a specific purpose. Figure 1B−D shows a schematic of three different OSDs used for AFM imaging. The CD may consist of five layers—labeling layer, protective layer, metal reflective layer, dye recording layer, and polycarbonate plastic layer at the bottom (Figure 1B). Similarly, the DVD may be composed of seven or more layers—label layer, top polycarbonate plastic layer, adhesive layer, protective layer, metal reflective layer, dye recording layer, and bottom polycarbonate layer (Figure 1C). Finally, the Blu-ray disc seemed to contain seven layers—label layer, polycarbonate plastic layer, metal reflective layer, dye recording layer, thin plastic cover layer, and hard coat layer (Figure 1D).

Procedure for Stripping the Protective Layers off Discs. The optical images of OSDs used in the lab can be found in Figure SI1 in the Instructor Notes. Detailed procedures for exposing the data storage layer for each OSD can be found in the Instructor Notes (Figure SI2).

Data Acquisition with AFM. Details about the AFM instrument and the parameters used can be found in "AFM Instrument and Parameters" section in the Instructor Notes. Briefly, the topographical measurements on the OSDs were performed in ScanAsyst tapping mode in air using a Bruker Dimension Icon V microscope controlled by NanoScope 9.7 software. The ScanAsyst air probe manufactured by Bruker used has a spring constant (k) of 0.4 N/m. The amplitude set point and peak force set point for all experiments were 250.55 mV and 15.03 nN, respectively.

Either Bruker's NanoScope-Analysis or Gwyddion software[33] was used to perform postprocessing data manipulation. The students were given choice of either NanoScope-Analysis or ImageJ[34] package for data manipulation and interpretation.

In general, the $F-d$ and $F-t$ plots are obtained using the tip deflection (nm) vs sample-tip separation (nm). Tip deflection (x) was converted into force by using Hooke's Law as follows:

$$F = kx \quad (1)$$

where F is force (nN) between the tip and sample, k is the spring constant of the tip (provided by the manufacturer), and

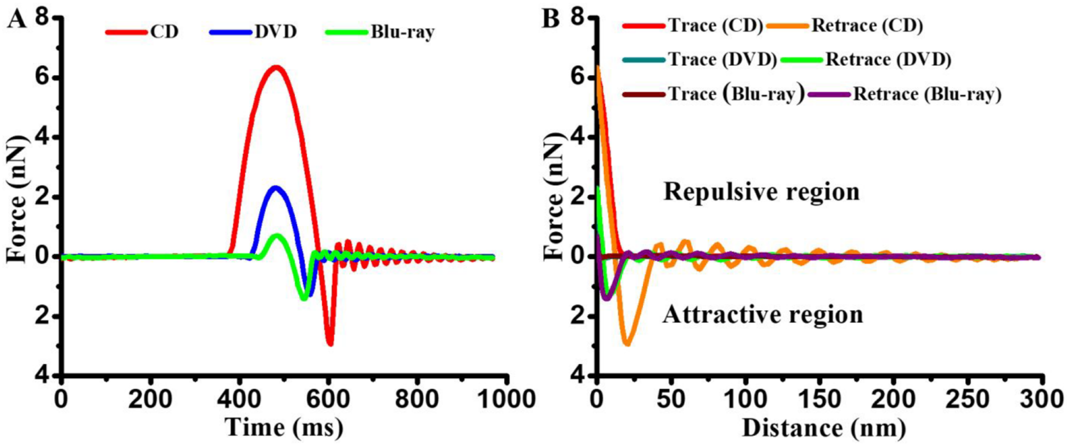


Figure 3. Tip−sample interaction curves for OSDs. (A) Average of three *F*−*t* curves. (B) Average of three *F*−*d* curves.

x is the tip deflection. The attractive and repulsive forces are usually represented with negative and positive signs, respectively.

Trace−Retrace Curves. This trace−retrace discussion is instructive for students to understand the working principles of the AFM and how *F*−*t* curves are converted into *F*−*d* curves. The trace−retrace curves indicate the direction of the cantilever movement with respect to the sample (Figure 2). The trace and retrace curves are directly correlated with the cantilever position and deflection of the piezoelectric head of the AFM where the tip is sitting. The application of appropriate voltage with a feedback loop from the controller accurately and precisely allows *F*−*t* and *F*−*d* measurements with a fraction of a nanometer (>30 pm) in the *z*-axis as well as in *x*- and *y*-axes (>150 pm in closed loop and >100 pm in open loop). For more information on hardware, please see Bruker's Web site to request a Dimension Icon Brochure.[35]

In the *F*−*t* curve, the trace starts from left and ends in the middle of the figure (red curve, Figure 2A), whereas the retrace starts in the middle and ends at the right (green curve, Figure 2A). On the other hand, in the *F*−*d* curve, the trace starts from the right and ends left (red curve, Figure 2B), whereas the retrace starts from the left and ends right of the curve (green curve, Figure 2B). The software derived *F*−*d* curves from *F*−*t* curves. The method of extracting an *F*−*d* curve from an *F*−*t* curve is discussed in the Detailed Materials and Methods, Supporting Information (see Data Analysis: Generating Force−Distance or Force−Time Curve from HSDC files subsection).

Generally, both the trace and retrace lines can be divided into three major segments. In *F*−*t* (Figure 2A) and *F*−*d* (Figure 2B) curves, the three trace segments are indicated by red arrows with a number assigned to them in a red bubble, whereas the retrace segments are indicated by green arrows with a number assigned to them in a green bubble. The corresponding tip position of those segments are shown in the insets. The black arrow in the insets indicate the up- or downward movement of the cantilever. Initially the cantilever is far from the sample where the tip−sample distance (d) > many hundreds of nanometers (inset 1 in Figure 2A and Figure 2B). A flat straight line is obtained when d is large enough such that there is almost no tip−sample interaction (segment 1 in red curves in Figures 2A and 2B). There is an attractive force between sample and tip showing a small dip in the trace line (segment 2 in red curve in Figures 2A and 2B). Enhanced electrostatic tip−sample repulsion (inset 3 in Figure 2A and Figure 2B) leads to a steeper *F*−*d* curve (segment 3 in red curves in Figures 2A and 2B). The maximum repulsive force (red square in Figure 2A and Figure 2B) in each experiment can be estimated at the inflection of red and green curves (Figure 2A and 2B).

Experimentally, the retraction of the tip off the sample surface is denoted by the retrace curve (green curves in Figure 2A and Figure 2B). The retrace curve can also be divided into three segments as a trace curve. After a maximum repulsive force is sensed by the feedback loop controller, it attempts to retract the tip away from the sample (inset 4 in Figure 2A and Figure 2B) reducing the sample-tip repulsive force (segment 4 in green curve in Figure 2B). The tip, however, may remain in contact with the sample surface due to attractive forces, which depends upon multiple factors including material properties of the tip and sample, and instrumental parameters as well.[36] Excessive applied force by the tip on the sample may damage the sample and/or tip—knowledge of material properties of the sample and tip may help in choosing appropriate instrumental parameters to avoid damage to the sample and tip. The cantilever overcomes the attractive force (inset 5 in Figure 2A and Figure 2B), freeing itself away from the sample (segment 5 in green curve in Figure 2A and 2B). This is the second segment of the retrace line. The cantilever may oscillate after leaving the sample surface (inset 6 in Figure 2A and Figure 2B) that shows up as sinusoidal (ringing) in the *F*−*t* and *F*−*d* curves (segment 6 in green curve in Figure 2B).[37]

During the generation of force curves, snap-in occurred as the tip transitioned from the air to the surface of the sample and snap-out occurred as the tip transitioned from the surface of the sample to the air.[38,39] The snap-in contact is observed from segment 1 to segment 2, whereas snap-out is observed in segment 5 to segment 6 (Figure 2A and Figure 2B). It should be noted the slopes of an *F*−*d* curve for segment 5 are smaller than that of segment 4. For example, the average *F*−*d* slope (n = 3) for segment 4 for CD samples is approximately three times larger than that of segment 5 (Table S3 in Detailed Materials and Methods, Supporting Information). These slope differences are due to much stronger tip−sample interactions for segments 3 and 4 than those for segments 2 and 5.[24]

Figure 3 shows the average *F*−*t* and *F*−*d* plots of three different spots of CD, DVD, and Blu-ray OSDs. The individual *F*−*d* and *F*−*t* curves for three different spots of CD, DVD, and Blu-ray are presented in Figure S1 in Detailed Materials and

https://doi.org/10.1021/acs.jchemed.2c00291
J. Chem. Educ. 2022, 99, 3030−3038

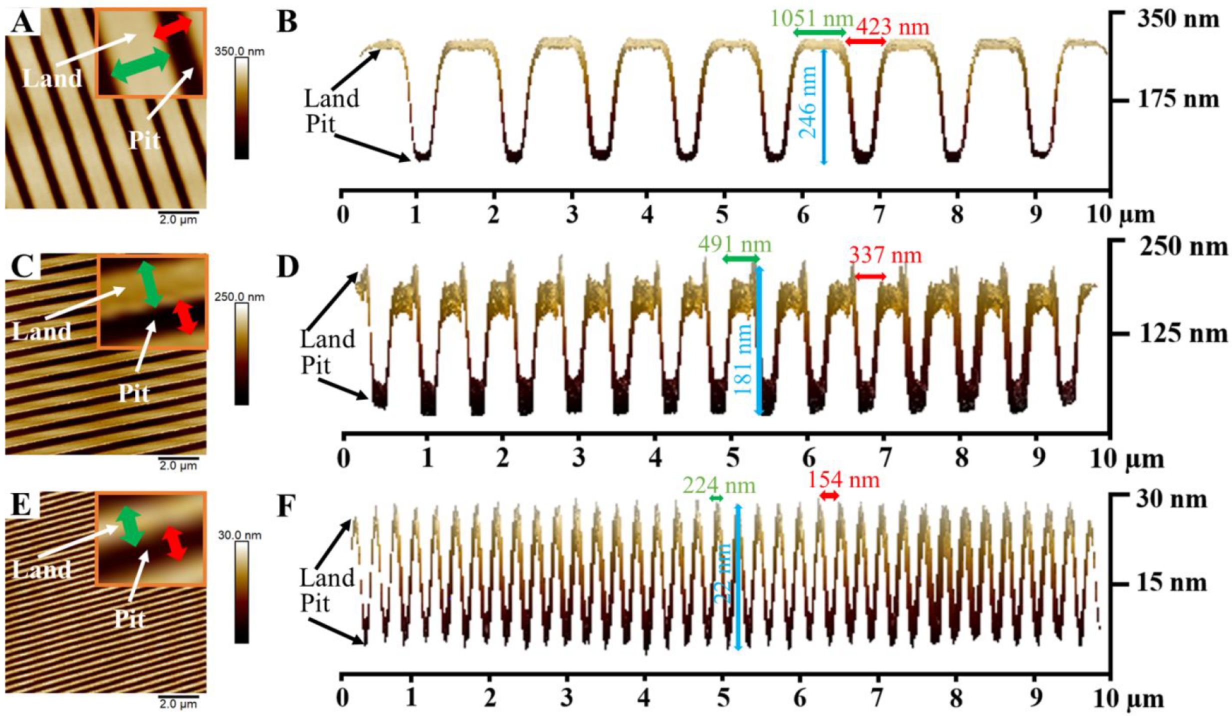


Figure 4. Representative topological images of OSDs. Top-view of the surface topographic of spot 1 of a CD (A), DVD (C), and Blu-ray (E) and corresponding side-view of spot 1 showing groove heights in B, D, and F, respectively. The height of the land is presented with a blue arrow; and the width of the land and width of the pit are shown with green and red arrows, respectively. The white arrows in A, C, and D indicate land and pits in inset with zoomed in images.

Methods, Supporting Information. The average measured repulsive forces of CD, DVD, and Blu-ray were 6.3 nN, 2.3 nN, and 0.7 nN, respectively, whereas their corresponding maximum attractive forces were 2.6 nN, 1.5 nN, and 1.7 nN, respectively. Similarity in force is likely because all the OSDs used in the present study are composed of the same polymer (polycarbonate) possessing similar mechanical properties. The detailed forces for each spot are presented in Table S2 in Detailed Materials and Methods, Supporting Information. There appear to be some differences in the magnitude of force in *F*−*t* and *F*−*d* curves. These differences are potentially due to the differences in physical and/or chemical properties of the building block of the samples and/or differences in their production parameters. Although CD, DVD, and Blu-ray OSDs are assumed to be composed of polycarbonate, they are fabricated by different companies and likely in different batches. Considering this, the physical properties (e.g., Young's modulus) are expected to be different for different types of OSDs, which is reflected in their *F*−*t* and *F*−*d* curves.

The *F*−*t* curves are sometimes referred to as a "heartbeat" and can be used to monitor real-time tip−sample interaction during a complete scanning cycle, which is translated into a topographic image by the response of feedback loop control.[40] At the same time, using *z*-position information on the tip, the "heartbeat" is transformed into a *F*−*d* curve by the software to produce many properties of interest including adhesion, modulus, deformation, dissipation, indentation, etc. if the samples are run in PF-QNM mode.[41] It is important to know that *F*−*t* curves can provide all properties of interest directly when analyzed, but presenting the *F*−*d* curves in the literature has remained a more common practice.

Representative AFM Images. The representative topological AFM images (top and side views of spot 1) of the CD, DVD, and Blu-ray are presented with the average width of the lands, average width of the pits, and average height of the lands (Figure 4). Figures S2 and S3 in the Detailed Materials and Methods (Supporting Information) represent the topological AFM images of spot 2 and spot 3, respectively. The estimated height and width of the lands, and width of the grooves/pits of three different spots of each OSDs are given in Table 2. More details for all three spots can be found in Table S1 in the Supporting Information. In general, the top flat portions of OSDs are defined as the lands. The grooves are valleys or channels of the OSDs. The flat lowest plane within the groove is called pit. The lands and grooves/pits are indicated in Figures 4, S2, and S3, showing top-view and side-view images of the OSDs.

Table 2. Average Width of Lands, Width of Pits, and Height Lands of CD, DVD, and Blu-ray

Sample	Land Width (nm) (std)	Pit Width (nm) (std)	Land/Pit Height (nm) (std)	Number of Measurements	Number of Spots
CD	1042 (±57)	450 (±29)	242 (±5)	7−15	3
DVD	491 (±17)	331 (±11)	184 (±4)	7−15	3
Blu-ray	226 (±13)	158 (±8)	23 (±1)	7−15	3

RESULTS AND DISCUSSION

Impact on Student Knowledge of AFM Components and Techniques

Prelab and Results: Part 1. All students participating in the study (*N* = 4) identified the key components of an AFM

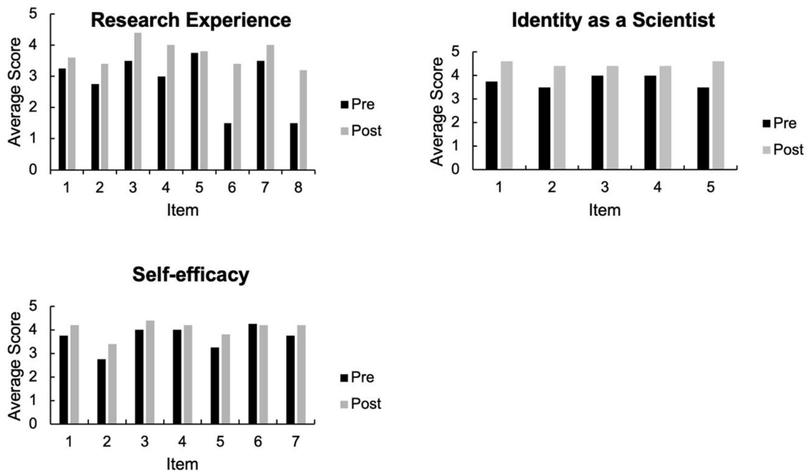


Figure 5. Comparison of student perceptions of research experience, self-efficacy, and identity-as-a-scientist prior to and after lab experiment completion.

such as the cantilever and tip, laser, photodiode, detector, and feedback electronics. Three of four students were able to associate these key components with an image of the Bruker Dimension Icon V AFM. Therefore, these elements of the investigation supported students' ability to both identify key generic AFM components and these components on a specific instrument. The prelab coupled with Part 1 of the investigation, subsequently, addressed a common shortfall of instrument-based courses where students obtain data from instruments, but have no knowledge of their internal workings.[42]

Results: Part 2. One student successfully placed the cantilever on the holder and mounted it in the scanhead, aligned the laser on the cantilever, and optimized the photodiode signal. They also successfully focused on an area of the sample by moving the scanhead with M.A.A.'s support. All groups obtained sufficient topographic data and force curves from their scans to complete their data analysis. Thus, with support, one student appropriately manipulated several of the key AFM components they identified to obtain acceptably accurate data. Most students, however, lacked the confidence to manipulate these components. The lab now includes a low stakes practice activity, with students in groups of two, to provide more hands-on training and operation of the AFM instrument to build confidence with AFM operational knowledge. Given cantilevers but without a tip—or if available with damaged tips—they mount the cantilevers on the probe holder and mount the tip probe on the scanhead with TA support. This latter task requires close attention from the students and TA because the scanhead is one of the most expensive parts of the instrument. Adopters of this experiment may consider creating a step-by-step video of this procedure suitable for their AFM for students to view as many times as needed prior to lab sessions.

Results: Part 3. A table to organize the mean measurements of grooves and plateaus was provided in the piloted lab. All students reported measurements of appropriate magnitude and significant figures in that table, but two of four did not include units of measurement. It was decided students should be more challenged in Part 3 by giving them the responsibility to decide how to organize their data. Subsequently, Part 3 of the Student Handout was modified by supplanting the provided data table with a prompt for students to construct their own table. We recommend giving students an opportunity to share ideas about table construction with a small group of peers either prior to and/or just after data collection.

Results: Part 4. Although students were able to correctly identify the spring constant range and dimensions of the cantilever from Bruker's Web site, two of four incorrectly identified the tip diameter as 650 nm as opposed to 4−24 nm. Students, consequently, may need more explicit guidance from the lab instructor/TA on how to interpret the manufacturer's provided information for their cantilevers and tips. Additionally, only two of four students described how cantilever and tip characteristics are integral to interpreting the obtained topographic and force curve data. Accordingly, Part 4 has been modified to now refer students back to the prelab references[23,24] to support their ability to connect the function of these key AFM components and nanoscale topographic data collection and interpretation.

Lab Data Analysis/Discussion

Two of the four students were also unable to estimate theoretical storage capacities and correctly match the unknown samples with the correct OSD type. Unsurprisingly, they also struggled to articulate a rationale for their matched choices despite the reference to match their data.[12] They seemed to struggle to use the Gwyddion software to complete their data analysis. Consequently, tutorial video links are now included in this section of the lab to support their ability to carry out data analysis using Gwyddion, and an instructional guide is provided to support their estimation of storage capacity (see Supporting Information).

Limitations

Students were able to identify plausible limitations of their data, but none specifically described how their identified limitations likely impacted the accuracy and precision of their obtained data. For example, some students stated sample preparation had the potential to damage disc surfaces. None, however, were then able to justify this limitation by connecting the degree of consistency across obtained topographic images and their 3D representations. Thus, despite being an advanced chemistry course, students were unable to connect why three scans from different regions were performed for each OSD sample during data collection; i.e., they did not consider the reliability of their measurements.

Extending Your Learning

Most students correctly described intermolecular repulsion forces of the chemically modified tip generating differences in electrical signals producing force curves for molecules as opposed to macroscopic samples such as OSDs. Thus, they were able to identify how one key component used in their investigation can be modified to produce structural data about a very different sample type.

Impact on Student Perceptions

To determine whether the lab potentially impacted student perceptions related to science, students' (N = 4) selected qualitative descriptors for each item on each scale were numerically ranked (a lot = 5, to a good extent = 4, to some extent = 3, a little = 2, not at all = 1; absolutely confident = 5, confident = 4, somewhat confident = 3, a little confident = 2, not at all confident = 1). Ranked selections for each item on each scale was aggregated and averaged. Figure 5 shows averaged item level ranked survey responses prior to and after students completed the lab. After completing the investigation, all participating students had increased positive perceptions of (1) how active they have been in research experiences, (2) their confidence or self-efficacy in completing science practices such as using scientific literature to guide research, and (3) how they think of their personal identity within science (see Supporting Information for survey items).

The relatively consistent increase across all items on all scales indicates the guided inquiry elements of the investigation improved—at least in the short term—how participating students affectively related to science. Some impact of other science courses on their affect toward the three constructs on the survey cannot be ruled out, but it is unlikely given the four-week time span in which the pre- and postsurveys were administered.

Conclusions

Responding to guidance from the ACS for undergraduate chemistry courses to engage students with nanoscale materials and use of pedagogical approaches guided by modern learning theories, we created, piloted, and revised a guided inquiry investigation centered around introducing them to key AFM components and basic data collection techniques. Specifically, students engaged with the structure and related storage capacities of three types of OSDs (CD, DVD, and Blu-ray). Students demonstrated knowledge of the internal workings of the AFM by identifying and using key components to obtain acceptable structural data. Some students struggled with using the research literature provided to match each unknown sample to one of the three OSD types and articulating reasons for their identified limitations. Yet, all participating students reported improvement on every survey item about their experiences with science research, ability to engage in science practices, and their personal scientific identity. This increase was a short-term observation, but it suggests transforming instrument-focused investigations to incorporate more inquiry-based features can improve students' knowledge of the instruments' inner workings, data collection and interpretation techniques, as well as their affect toward science.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00291.

- Instructor Notes (PDF) (DOCX)
- Raw Data Files (ZIP)
- Student Handout (PDF) (DOCX)
- Detailed Materials and Methods (PDF) (DOCX)
- Estimation of OSD Storage Capacity Guide (PDF) (DOCX)
- Survey Instrument (PDF) (DOCX)

■ AUTHOR INFORMATION

Corresponding Author

Senetta F. Bancroft − *School of Education, Southern Illinois University Carbondale, Carbondale, Illinois 62901, United States; School of Chemical and Biomolecular Sciences, Southern Illinois University Carbondale, Carbondale, Illinois 62901, United States;* orcid.org/0000-0002-7498-1169; Email: senetta.bancroft@siu.edu

Authors

M. Aswad Ali − *School of Chemical and Biomolecular Sciences, Southern Illinois University Carbondale, Carbondale, Illinois 62901, United States*

Punit Kohli − *School of Chemical and Biomolecular Sciences, Southern Illinois University Carbondale, Carbondale, Illinois 62901, United States;* orcid.org/0000-0003-3183-4385

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.jchemed.2c00291

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Funding from the NSF MRI Grant No. CHE-1920255 is acknowledged for supporting the purchase of the AFM instrument. We also acknowledge the School of Chemical and Biomolecular Sciences and Office of Vice-Chancellor of Research (OVCR) at SIUC for supporting the AFM instruments and supplies used in the experiments.

■ REFERENCES

(1) American Chemical Society. *Undergraduate Professional Education in Chemistry: ACS Guidelines and Evaluation Procedures for Bachelor's Degree Programs*; American Chemical Society: Washington, D.C., 2015.

(2) Parker, A. K.; Danielson, E. T.; Black, S. D. Nanochemical Activity for Undergraduates with ChemToy2. *J. Chem. Educ.* **2021**, *98* (9), 2944−2951.

(3) Trought, M.; Perrine, K. A. Investigating the Relationship between Adhesion Forces and Surface Functionalization Using Atomic Force Microscopy. *J. Chem. Educ.* **2021**, *98* (5), 1768.

(4) Ashkenaz, D. E.; Hall, W. P.; Haynes, C. L.; Hicks, E. M.; McFarland, A. D.; Sherry, L. J.; Stuart, D. A.; Wheeler, K. E.; Yonzon, C. R.; Zhao, J.; Godwin, H. A.; Van Duyne, R. P. Coffee Cup Atomic Force Microscopy. *J. Chem. Educ.* **2010**, *87* (3), 306−307.

(5) Sanders, W. C.; Ainsworth, P. D.; Archer, D. M.; Armajo, M. L.; Emerson, C. E.; Calara, J. V.; Dixon, M. L.; Lindsey, S. T.; Moore, H. J.; Swenson, J. D. Characterization of Micro- and Nanoscale Silver Wires Synthesized Using a Single-Replacement Reaction between

Sputtered Copper Metal and Dilute Silver Nitrate Solutions. *J. Chem. Educ.* **2014**, *91* (5), 705−710.

(6) Ito, T. Observation of DNA Molecules Using Fluorescence Microscopy and Atomic Force Microscopy. *J. Chem. Educ.* **2008**, *85* (5), 680−682.

(7) Binnig, G.; Despont, M.; Drechsler, U.; Häberle, W.; Lutwyche, M.; Vettiger, P.; Mamin, H. J.; Chui, B. W.; Kenny, T. W. Ultrahigh-Density Atomic Force Microscopy Data Storage with Erase Capability. *Appl. Phys. Lett.* **1999**, *74* (9), 1329−1331.

(8) Jo, A.; Joo, W.; Jin, W. H.; Nam, H.; Kim, J. K. Ultrahigh-Density Phase-Change Data Storage without the Use of Heating. *Nat. Nanotechnol.* **2009**, *4* (11), 727−731.

(9) Lantz, M. A.; Wiesmann, D.; Gotsmann, B. Dynamic Superlubricity and the Elimination of Wear on the Nanoscale. *Nat. Nanotechnol.* **2009**, *4* (9), 586−591.

(10) Pivovarov, P. A.; Zavedeev, E. V.; Frolov, V. D.; Roch, T.; Scheibe, H. J.; Pimenov, S. M. Atomic-Force-Microscopy Nano-writing on Ultrathin Tetrahedral Amorphous Carbon Films. *Appl. Phys. A Mater. Sci. Process.* **2016**, DOI: 10.1007/s00339-016-0509-6.

(11) Marques, I. H. G.; Saraiva Matos, R.; Romaguera-Barcelay, Y.; Ţălu, Ş.; Moreira, J. A.; Almeida, A.; De Cruz, J. P.; Da Fonseca Filho, H. D. Investigation of Stereometric and Fractal Patterns of Spin-Coated LuMnO3Thin Films. *Adv. Mater. Sci. Eng.* **2021**, *2021*, 1.

(12) Hwu, E. E. Te; Boisen, A. Hacking CD/DVD/Blu-Ray for Biosensing. *ACS Sens.* **2018**, *3* (7), 1222−1232.

(13) Márquez, A.; Álvarez, M. L.; Neipp, C.; Méndez, D.; Beléndez, A. Effective Physical Optics Hands-on Experience through the Characterization of a CD and a DVD. *Proc. SPIE* **2005**, *9664*, 96642L.

(14) Bächtold, M. What Do Students "Construct" According to Constructivism in Science Education? *Res. Sci. Educ.* **2013**, *43* (6), 2477−2496.

(15) National Research Council. *A Framework for K-12 Science Education: Practices, Crosscutting Concepts, and Core Ideas*; National Academy Press: Washington, D.C., 2012.

(16) Bruck, L. B.; Bretz, S. L.; Towns, M. H. Characterizing the Level of Inquiry in the Undergraduate Laboratory. *J. Coll. Sci. Teach.* **2008**, *38* (1), 52−58.

(17) Koedinger, K. R.; Corbett, A. T.; Perfetti, C. The Knowledge-Learning-Instruction Framework: Bridging the Science-Practice Chasm to Enhance Robust Student Learning. *Cogn. Sci.* **2012**, *36* (5), 757−798.

(18) Larson, K. G.; Long, G. R.; Briggs, M. W. Periodic Properties and Inquiry: Student Mental Models Observed during a Periodic Table Puzzle Activity. *J. Chem. Educ.* **2012**, *89* (12), 1491−1498.

(19) Bodemer, D.; Faust, U. External and Mental Referencing of Multiple Representations. *Comput. Human Behav.* **2006**, *22* (1), 27−42.

(20) Rau, M. A. Making Connections among Multiple Visual Representations: How Do Sense-Making Skills and Perceptual Fluency Relate to Learning of Chemistry Knowledge? *Instr. Sci.* **2018**, *46* (2), 209−243.

(21) Taber, K. S. Revisiting the Chemistry Triplet: Drawing upon the Nature of Chemical Knowledge and the Psychology of Learning to Inform Chemistry Education. *Chem. Educ. Res. Pract.* **2013**, *14* (2), 156−168.

(22) Kararo, A. T.; Colvin, R. A.; Cooper, M. M.; Underwood, S. M. Predictions and Constructing Explanations: An Investigation into Introductory Chemistry Students' Understanding of Structure-Property Relationships. *Chem. Educ. Res. Pract.* **2019**, *20* (1), 316−328.

(23) Binnig, G.; Quate, C. F.; Gerber, C. Atomic Force Microscope '. *Phys. Rev. Lett.* **1986**, *56* (9), 930−933.

(24) Butt, H. J.; Cappella, B.; Kappl, M. Force Measurements with the Atomic Force Microscope: Technique, Interpretation and Applications. *Surf. Sci. Rep.* **2005**, *59* (1−6), 1−152.

(25) Han, T.; Williams, J. M.; Beebe, T. P. Chemical Bonds Studied with Functionalized Atomic Force Microscopy Tips. *Anal. Chim. Acta* **1995**, *307* (2−3), 365−376.

(26) Liebig, A.; Hapala, P.; Weymouth, A. J.; Giessibl, F. J. Quantifying the Evolution of Atomic Interaction of a Complex Surface with a Functionalized Atomic Force Microscopy Tip. *Sci. Rep.* **2020**, *10* (1), 14104.

(27) Uchihashi, T.; Iino, R.; Ando, T.; Noji, H. High-Speed Atomic Force Microscopy Reveals Rotary Catalysis of Rotorless F 1-ATPase. *Science* **2011**, *333* (6043), 755−758.

(28) Gross, L.; Mohn, F.; Moll, N.; Liljeroth, P.; Meyer, G. The Chemical Structure of a Molecule Resolved by Atomic Force Microscopy. *Science* **2009**, *325* (5944), 1110−1114.

(29) Gross, L.; Mohn, F.; Moll, N.; Schuler, B.; Criado, A.; Guitián, E.; Peña, D.; Gourdon, A.; Meyer, G. Bond-Order Discrimination by Atomic Force Microscopy. *Science* **2012**, *337* (6100), 1326−1329.

(30) Zhang, J.; Chen, P.; Yuan, B.; Ji, W.; Cheng, Z.; Qiu, X. Real-Space Identification of Intermolecular Bonding with Atomic Force Microscopy. *Science* **2013**, *342* (6158), 611−614.

(31) de Oteyza, D. G.; Gorman, P.; Chen, Y.-C.; Wickenburg, S.; Riss, A.; Mowbray, D. J.; Etkin, G.; Pedramrazi, Z.; Tsai, H.-Z.; Rubio, A.; Crommie, M. F.; Fischer, F. R. Direct Imaging of Covalent Bond. *Science* **2013**, *340*, 1434−1437.

(32) Robnett, R. D.; Chemers, M. M.; Zurbriggen, E. L. Longitudinal Associations among Undergraduates' Research Experience, Self-Efficacy, and Identity. *J. Res. Sci. Teach.* **2015**, *52* (6), 847−867.

(33) Nečas, D.; Klapetek, P. Gwyddion: An Open-Source Software for SPM Data Analysis. *Open Phys.* **2012**, *10* (1), 181−188.

(34) Rasband, W. S. *Image J*; U.S. National Institutes of Health: Bethesda, MD, 2018.

(35) Dimension Icon | Bruker, https://www.bruker.com/en/meta/forms/bns-form-pages/brochures/afmi/dimension-icon.html (accessed on March 21, 2022).

(36) Magonov, S. Visualization of Polymers at Surfaces and Interfaces with Atomic Force Microscopy. In *Handbook of Surfaces and Interfaces of Materials*; Elsevier, 2001; pp 393−430.

(37) Eppell, S. J.; Li, L.; Zypman, F. R. Unexplored Territory in the AFM Force Curve Contains Nanomechanics Information. *AIP Adv.* **2017**, 7 (10), 105208.

(38) Hutter, J. L.; Bechhoefer, J. Calibration of Atomic-force Microscope Tips. *Rev. Sci. Instrum.* **1993**, *64* (7), 1868.

(39) Zhao, B.; Song, Y.; Wang, S.; Dai, B.; Zhang, L.; Dong, Y.; Lü, J.; Hu, J. Mechanical Mapping of Nanobubbles by PeakForce Atomic Force Microscopy. *Soft Matter* **2013**, *9* (37), 8837−8843.

(40) Foster, B. New Atomic Force Microscopy(AFM) Approaches Life Sciences Gently, Quantitatively, and Correctively. *Am. Lab.* **2012**, *44* (4), 24−28.

(41) Pittenger, B.; Erina, N.; Su, C.; Ohler, B. Quantitative Mechanical Property Mapping at the Nanoscale with PeakForce QNM. *Wiley Analytical Science Magazine*, December, 2010. https://analyticalscience.wiley.com/do/10.1002/micro.152 (accessed on June 24, 2022).

(42) National Research Council. Instructional Strategies. *Discipline-Based Education Research: Understanding and Improving Learning in Undergraduate Science and Engineering*; National Academies Press: Washington, D.C., 2012; pp 119−139.

https://doi.org/10.1021/acs.jchemed.2c00291
J. Chem. Educ. 2022, 99, 3030−3038