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Three-Dimensional Printing of a Model Atomic Force Microscope to Measure Force−Distance Profiles

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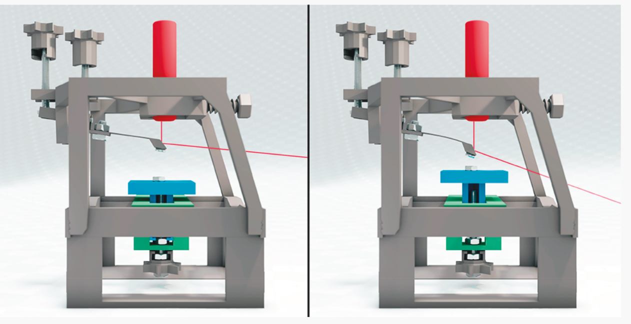

ABSTRACT: We report a simple means to build a model atomic force microscope (AFM) using 3D printing of thermoplastic materials that are commercially available. The model has many of the key parts of an actual AFM including a *z*-axis stage, an AFM head with a cantilever assembly, and a laser source that reflects off of the back of the cantilever. Using a magnet attached to the tip of the cantilever and a metal sample, this model AFM enables acquisition of force−distance profiles with characteristic snap-in, pull-off, separate, and contact regions. The model AFM was designed, printed, and used by first- and second-year undergraduate students. Through completion of this project, students learned scientific instrument design and construction via 3D printing and obtained first-hand practice in the measurement of force−distance profiles and the elastic constants of cantilevers. The open design of the model can easily accommodate additional capabilities in which students are interested, e.g., topographical scanning and using cantilevers made from different materials.

KEYWORDS: *First-Year Undergraduate/General, Analytical Chemistry, Physical Chemistry, Laboratory Instruction, Analogies/Transfer, Hands-On Learning/Manipulatives, Applications of Chemistry, Nanotechnology, Surface Science, Undergraduate Research*

INTRODUCTION

Atomic force microscopy has been a useful tool in chemistry and materials science due to its exquisitely high spatial resolution that has been used to visualize materials, molecules, and atoms.[1,2] However, using an atomic force microscope (AFM) in the laboratory curriculum is typically restricted to upper-division undergraduate or higher-level courses.[3−5] At the lower-division undergraduate level, AFM use is uncommon and generally restricted to laboratories with fewer than 10 students[6−8] or to use of the AFM as a demonstration during class.[9] To understand and take full advantage of its technical capabilities, an understanding of an AFM's operating principles is necessary, and in our view, should start at the first-year undergraduate level.

Building and using model AFMs has proven to be effective for educational purposes at the undergraduate level.[10−21] There have been simple, inexpensive models for which groups of students could collect data without expert supervision.[10−13,19] One example of these simple models was a coffee cup model AFM that was used to perform a line scan with a height resolution of ∼0.1 mm.[10] While a majority of the model AFMs have focused on "scanning" in the plane of the surface, two models have collected data by moving the sample along the surface normal to produce force−distance profiles.[16,21] For each of these models, force was measured directly (using a type of force sensor) as a function of distance between two magnets in the non-contact region. We report a model AFM that was used to experimentally determine force−distance profiles that did model the snap-in, pull-off, separate, and contact regions associated with these profiles. In addition, our model was designed, modified, and 3D printed by first- and second-year undergraduate students.

Our approach utilized 3D printing of commercially available thermoplastic materials. With the wide availability of 3D printers, the material cost was less than $10 per model AFM, and the 3D object files were easily passed to new groups of students for modification and printing. Our model AFM mimicked the movement of an actual AFM with a *z*-axis stage that allowed movement of the sample along the surface normal. A laser pointer was added to model the "deflection configuration" of an AFM as the laser beam was reflected off the back of a cantilever and onto a surface for recording. As reported below, this simple model AFM enabled acquisition of force−distance profiles with characteristic snap-in, pull-off,

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separate, and contact regions. The open design easily accommodated additional capabilities in which students were interested including designing and 3D printing an *xy*-stage and incorporating cantilevers of various thicknesses.

■ CONSTRUCTION OF THE MODEL AFM AND ACQUSITION OF FORCE–DISTANCE PROFILES

We designed all parts of the model AFM using SketchUp (www.sketchup.com) and Blender (www.blender.org) and then exported the designs in STL format to be printed via a 3D printer. The STL files were included in the Supporting Information (SI). While various 3D printers could be used in printing our design, our printer was a Replicator 2 (Makerbot, Brooklyn, NY). Our model AFM (Figure 1) had two main parts: the base and the "AFM head". The base consisted of a structure into which the *z*-axis stage fit and onto which the AFM head sat. Assembly was relatively easy, but we also included detailed assembly instructions and a cost analysis in the Supporting Information for interested readers. To achieve submillimeter mechanical movement precision, we used standard steel 6-32 screws which are available at most hardware stores.

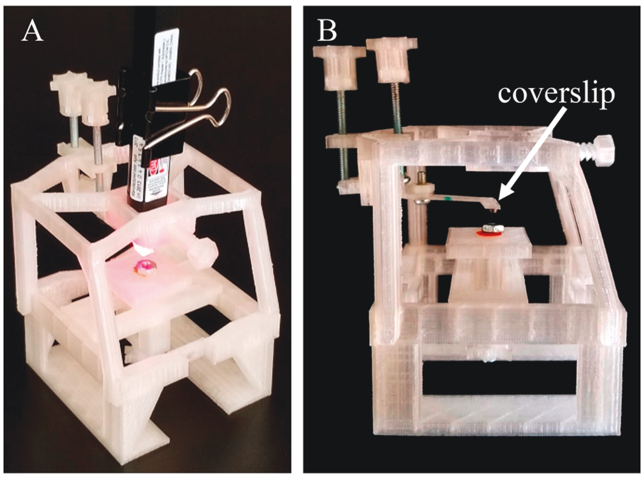

Figure 1. Photos of the model AFM with the *z*-axis stage and the cantilever. A neodymium magnet was glued onto the tip of the cantilever and a 6-32 nut was used as the sample: (A) with a laser pointer reflecting off of the piece of coverslip on the end of the cantilever and (B) a side view with the coverslip glued to the end of the cantilever.

The AFM head incorporated a cantilever assembly and a laser pointer (MP-1200Q, Quartet, ACCO Brands, Lake Zurich, IL). The cantilever (middle beam) was attached to the AFM head by a screw and had two other positioning screws that allowed the height and angle of the cantilever to be adjusted. The probe end of the cantilever had a flat surface onto which a neodymium magnet (D21B-N52, K&J Magnetics, Pipersville, PA) was superglued. A piece of either a coverslip or a microscope slide was also superglued onto the angled side of the probe end of the cantilever. This glass material reflected the laser beam out of the front of our model AFM. The position of the reflected laser was typically monitored using 6–7 pieces of graph paper positioned vertically on a wall approximately 1 m away.

Recording a Force–Distance Curve

The model AFM was set up to record a force–distance curve by attaching a neodymium magnet to the tip of a 3D printed cantilever and using a steel 6-32 nut as the sample. The steps to obtain a force–distance curve were the following:

1. Record position of the laser versus number of turns of the screw
2. Record position of the laser versus mass loaded on the tip of the cantilever
3. Create a plot of position of the laser versus *z*-axis movement
4. Estimate the magnification (gain) factor
5. Estimate the spring constant of the cantilever
6. Plot the force–distance profile

1. Record the Position of the Laser versus Number of Turns of the Screw. The *z*-axis stage was placed at its lowest position. The cantilever was adjusted to a position such that there was essentially no attraction between the magnet on the tip of the cantilever and the metal sample. The stage was then raised by turning the *z*-axis screw one turn at a time so that the sample approached the tip of the cantilever. With each turn, the position of the laser (on the graph paper on the wall) was recorded. The stage was raised until the cantilever tip jumped into contact with the sample after which 5–6 more points were recorded with the tip of the cantilever in contact with the sample (the contact region). The sample was then retracted from the tip to its original starting position one turn at a time. At the point for which there were significant movements of the cantilever tip as evidenced by the change in the position of the laser, we allowed extra time for the position of the cantilever (and, therefore, position of the laser) to equilibrate. This was especially true when retracting the stage.

2. Record the Position of the Laser versus Mass Loaded on the Tip of the Cantilever. With the stage at its lowest point (or with the AFM head removed completely from the base), the tip/cantilever was in its free state, far away from surfaces. Then, a series of metal objects of known mass, such as 6-32 nuts, were sequentially placed onto the magnetic tip. After each object was added, the position of the laser was recorded and then plotted against the mass added.

3. Create a Plot of Position of the Laser versus *z*-Axis Movement. The screws used in the model AFM were standard 6-32 screws with a well-defined number of turns per inch (32), equivalent to 0.79 mm/turn. Using this conversion factor, turns of the screw were converted to movement along the *z*-axis. The data from step 1 were then replotted as position of the laser versus *z*-axis movement.

4. Estimate the Magnification (Gain) Factor. To estimate the magnification (gain) factor, only the data from the contact region was used. The data from step 3 were replotted for the approach and retraction. In the case of contact, the *z*-axis movement was equal to the cantilever bending, in principle. Therefore, both the approach and the retraction data should be linear with the same slope. A least-squares fit was applied for both approach and retraction plots, and the average of the two values of the slope was taken. The slope of the trendline was the magnification (gain) factor, i.e., movement of laser on the wall/movement of cantilever along the *z*-axis.

5. Estimate the Spring Constant of the Cantilever. Starting with the plot of position of the laser versus mass loaded on tip of cantilever (step 2), the position of the laser

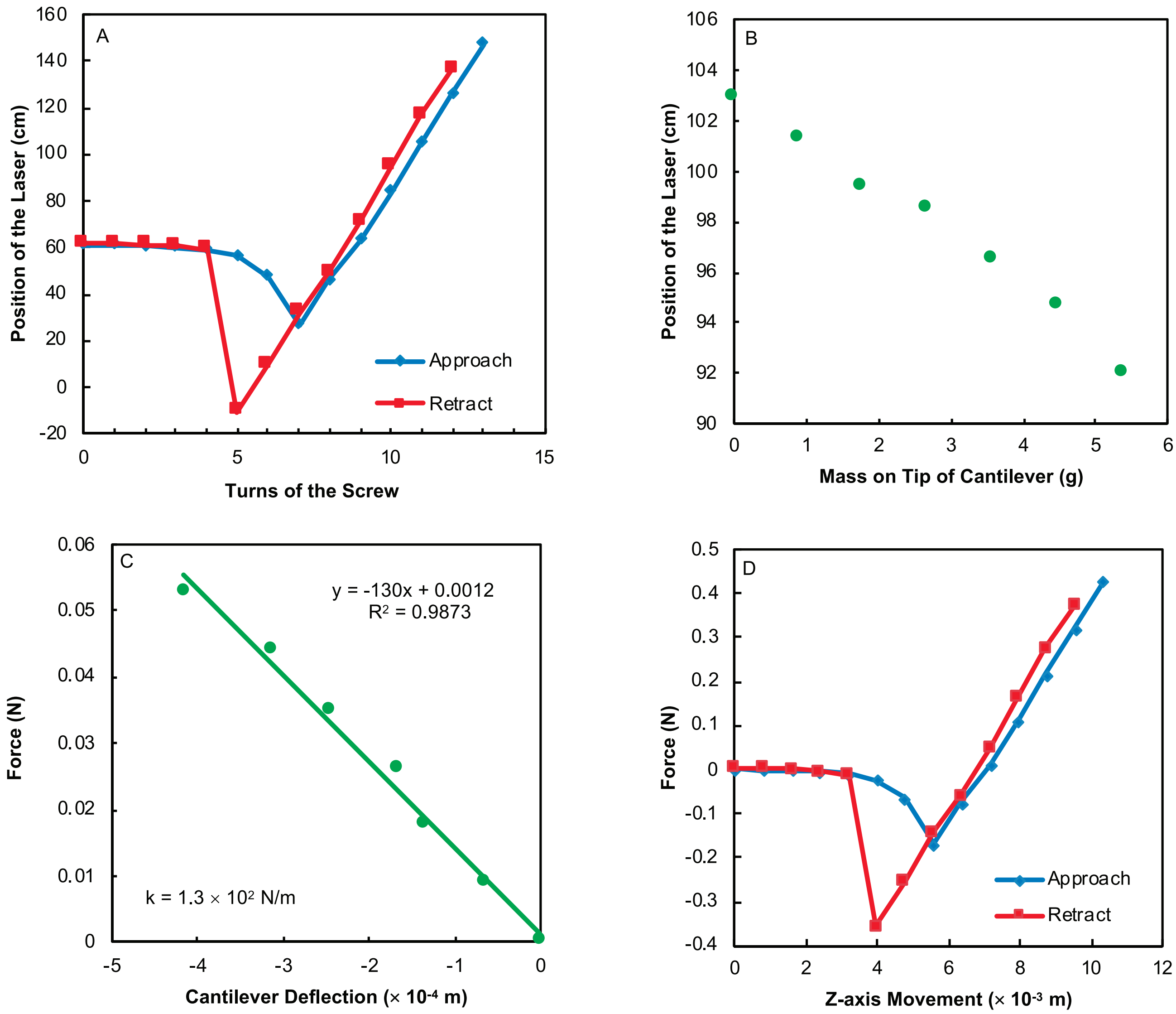

Figure 2. Data and analysis for the model using the *z*-axis stage and a 0.8 mm thick 3D printed poly(lactic acid) cantilever with a neodymium magnet glued to the tip of the cantilever and a 6-32 steel nut as a sample. For this experiment, the target was 1.66 m away from the model AFM. The magnet affixed to the cantilever was a model D21B-N52 from K&J Magnetics with a pull force of 0.56 pounds, a diameter of 1/8 in., and a thickness of 1/16 in. (A) Raw data: plot of position of the laser on the target versus number of turns of the screw with a 6-32 nut as a sample. (B) Raw data: position of the laser versus mass added to tip of cantilever. The masses were 6-32 nuts. (C) Processed data: force versus cantilever deflection. (D) Processed data: force versus distance profile. Force was set to zero at a *z*-axis movement of zero.

was converted to the movement of cantilever using the magnification factor extracted from step 4. Then, the gravitational constant was used to convert mass (in kg) into force (in N) ($F = mg$). Finally, the force was plotted versus *z*-axis movement, which followed a linear relationship. Assuming Hooke's law was valid for the bending of the cantilever

$$k = F/\Delta Z \tag{1}$$

where F was the force (in N) and ΔZ was the movement of the cantilever (in m) along *z*-axis, and then k was the spring constant of the cantilever (in N/m).

6. Plot the Force–Distance Profile. Starting with the plot of position of the laser versus *z*-axis movement from step 3, the position of the laser was converted into force using the magnification factor (step 4) and k (step 5),

$$F = (\text{position of the laser})/(\text{magnification factor}) \times k \tag{2}$$

The force at the first data point was set to zero, and the data was replotted as force versus *z*-axis movement.

RESULTS AND DISCUSSION

Figure 2A is a plot of the raw data collected from step 1 using our model AFM, while Figure 2B shows a plot of the raw data collected from step 2. Figure 2C is the result from step 5 from which the cantilever spring constant is extracted from the slope, $k = 1.3 \times 10^2$ N/m. The linear relationship is valid, as demonstrated by $R^2 = 0.9873$ in Figure 2C. Figure 2D is the final outcome following step 6, where a complete force–distance profile is shown. By using a magnetic tip and metal sample, the characteristics of an actual AFM's force–distance profile, such as the "snap-in" effect on approach and the "pull-off" effect on retraction, are clearly shown. In actual AFM, these effects are due to van der Waals and capillary forces, respectively.[1]

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Our students performed over one dozen measurements and identified two possible sources of error that could impact the reproducibility of the cantilever deflection during data acquisition. First, there were errors associated with the turning of the screw to raise and lower the stage. These fell into two categories: (a) a sinusoidal undulation in the position of the stage associated with less than full turns and (b) a hysteresis in the position of the screw when the stage was retracted. When students collected data at intervals of 1/7th of a turn (0.11 mm), they noticed a sinusoidal undulation in the position of the laser. Students resolved this by only collecting data for complete turns of the screw. For the hysteresis, students practiced turning the screw in one direction during approach and another direction during retreat. Mixed movement, e.g., one turn clockwise and then a half-turn counterclockwise, was avoided. Second, there were errors associated with the relaxation of the cantilever under the influence of the magnetic fields. This effect became more significant when the cantilever was bent to a higher degree: during the snap-in and pull-off regions. However, the students found that waiting for 3−5 min was generally sufficient for the cantilever to end its relaxation. For experiments with thinner, more flexible cantilevers, relaxation of the cantilever required up to 30 min.

This report represents the first 3D printed model AFM that enabled force−distance profiles in the contact region and whose design, construction, and practice were completely executed by first- and second-year undergraduate students. Students not only learned about scientific instrument design and construction via 3D printing but also developed their interests in future modifications of the model and attained first-hand practice in its utilization.

Students inserted a cantilever into the model AFM and positioned the cantilever above the sample stage at the correct height to record an image. They also aligned the laser pointer to make sure that its beam reflected off of the surface attached to the back of the cantilever (piece of coverslip or glass slide) and onto a flat surface. Although not the most optimal way for imaging, students learned many of the concepts of scanning probe microscopy.

Students learned to measure a true force−distance profile and measured the elastic constants for their own cantilevers. They compared force−distance profiles from various cantilevers they printed and compared the similarity and differences in "snap-in" and "pull-off" effects when cantilever thicknesses were changed. In doing so, they understood force profiles observed in actual AFM publications. Students also got a chance to practice the data analysis required to convert raw data into a property of their cantilever (spring constant) and a property of their surface (force versus distance). This is the first report of a simple model of an AFM that can be used by students to acquire true force−distance profiles qualitatively and quantitatively.

■ CONCLUSION

This report introduced a simple means to build a model AFM using 3D printing. The model had the key parts of the actual AFM, e.g., z-axis stage, an AFM head with a cantilever assembly, and a laser source using a laser pointer. Using a magnet attached to the tip of the cantilever and metal samples, this model AFM enabled acquisition of force−distance profiles, with characteristic snap-in, pull-off, separate, and contact regions.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.9b01099.

3D printed macroscale AFM model build instructions (PDF, DOCX)

Cost matrix for model AFM (XLSX)

Files for 3D printing the model of an AFM: half AFM top, half AFM stage, and AFM z-axis stage (ZIP)

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Notes

The authors declare no competing financial interest.

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