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A Macroscale Model for Hands-On Activities Demonstrating Transmission Electron Microscopy

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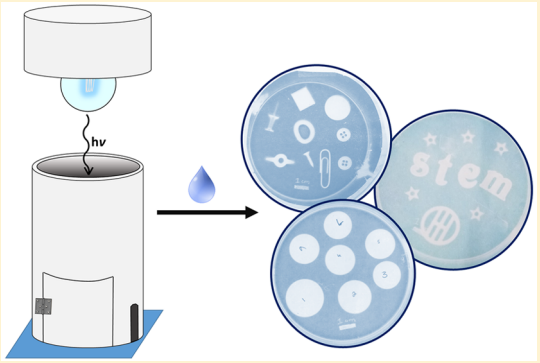
Supporting Information

ABSTRACT: Although nanotechnology lessons are increasingly integrated into curricula, students still face significant challenges in understanding the characterization techniques used to investigate nanotechnology. Many characterization techniques, such as transmission electron microscopy (TEM), are prohibitively expensive for primarily undergraduate institutions and completely inaccessible for high schools and local outreach programs. When TEMs are accessible, opportunities for hands-on use are still limited due to logistics and costs for instrument time. In this project, we present a low cost ($50 USD) macroscale TEM model that uses cyanotype paper for "imaging" and is constructed of materials available at local hardware and pet supply stores and chemistry stockrooms. This model allows students to investigate properties of TEM micrographs including thickness contrast, diffraction contrast, plan view, and tilt series imaging through a series of four hands-on activities in an active-learning setting. The four activities are identification of mystery objects from student cyanotype "micrographs", sizing objects from their micrographs using a scale bar, sketching the structure of a mystery 3D object from acquired tilt series images, and developing unique micrographs with objects of the students' choice and predicting features of the resulting images. The TEM model and associated activities were tested with groups of STEM (Science, Technology, Engineering and Mathematics)-interested high schoolers and were found to enhance student engagement, enjoyment, and understanding of certain properties of TEM micrographs.



KEYWORDS: *Nanotechnology, First-Year Undergraduate/General, Demonstrations, Hands-On Learning/Manipulatives, Materials Science, Analogies/Transfer, Instrumental Methods*

INTRODUCTION

As the field of nanotechnology continues to grow in academic and industrial settings, there are increasing efforts to introduce nanotechnology into chemistry curricula. Nanotechnology presents distinct challenges to incorporation into the curriculum. In particular, the difficult-to-visualize scale of nanotechnology and the breadth of complex instrumental techniques required to investigate nanomaterials result in challenges unique to this field.[1] Despite these challenges, there are several activities that incorporate nanotechnology into curricula at educational levels as young as pre-high school.[2−5]

To engage students in the topic of nanotechnology, it is necessary to include education about the tools used to investigate nanomaterials such as electron microscopy and scanning probe microscopy, among others. However, introducing these materials characterization methods presents its own challenges. Electron microscopes are unlikely to be found on primarily undergraduate campuses despite the growing importance of introducing such characterization methods to undergraduate cohorts,[6] so instrument availability is a factor. There have been some successful outreach programs that allow students to interact with instrumentation up close and for prolonged periods[7,8] as well as several programs where remote access is an option.[9,10] While there is high value in introducing students to instruments, as demonstrated by these programs, these are still cost- and class-size-limited solutions. To allow students to experiment with the key concepts of materials characterization techniques without instrumental access, several macroscale models of relevant instrumentation have been described in the literature.[11−15] Of these models, most are scanning probe microscopy (SPM), with a focus on atomic force microscopy.[11,14,15] Despite these exciting precedents, there are few options for modeling electron microscopy. To address this need, we developed an inexpensive macroscale model of a transmission electron microscope (TEM) that produces plan view "micrographs" on cyanotype paper and is capable of a tilt series function.

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Transmission electron microscopy is often used for characterization of nanomaterials as well as fixed and embedded biological samples such as bacteria or blood platelets.[16] For characterization of nanomaterials, TEM is important because it is possible to achieve higher spatial resolution than can be obtained with scanning electron microscopy (SEM), making it a workhorse technique of nanotechnology research. Despite the ubiquity of TEM in nanotechnology literature, introducing it to students is challenging due to (1) lack of instrument availability, (2) instrument use cost, (3) the amount of time involved in becoming skilled in electron microscopy, and (4) level of spatial reasoning needed for analysis of TEM micrographs. Point (4) can be particularly challenging in contrast to SEM, as it is not practical to train students on TEM instrumentation to develop skills to understand micrographs. Despite these difficulties, exposing students to TEM would encourage a greater understanding of micrographs and the resulting data they will encounter in the literature.

We have developed a model that demonstrates the following critical aspects of transmission electron microscopy: (a) 2D plan view images, (b) monochromatic output images, (c) "mass" (opacity) contrast, (d) thickness contrast, (e) pseudodiffraction contrast, and (f) images produced with scale bars for post-analysis. This is achieved by pairing the photographic printing process of cyanotyping with a modified PVC model of a TEM that was inspired by a model presented in the NanoDays Kit developed by NISENet.[17] The NISENet model uses PVC piping to represent the body of the TEM and a flashlight to represent the electron beam. In the NISENet activity, students determine the shapes of objects in the "sample stage" by relating them to the projected shadow. This demonstration illustrates the plan view characteristic of TEM micrographs but does not model features (b–f) and is very sensitive to room lighting.[17] Inspired by the base idea of the NanoDays Kit, we constructed a similar device to achieve goals (b–f) through the addition of a stage that can be rotated, cyanotyping paper for projections, and a UV-light source to activate the cyanotyping paper.

Cyanotyping, used here for its ability to produce 2D images with mass (or opacity) and thickness contrast, is a photographic printing process that produces prints by way of fixation of insoluble Prussian Blue (iron (II,III) hexacyanoferrate (II, III)) after reaction with UV light.[18] Historically, cyanotypes were used for producing architectural blueprints and are notable as being the medium Anna Atkins used to create prints of British ferns and algae in 1843.[18] Cyanotypes are popular in high school photography and art classes because they have a quick processing time under sunlight (1–5 min without cloud cover) and only need water for fixation.[19] Because fixation of Prussian blue is proportional to UV light exposure, cyanotypes are sensitive to shadows, semitransparent items, and the focus of light through objects such as glass marbles. This allows for the creation of stable prints that have properties analogous to mass, thickness, and diffraction contrast, which students can analyze for the activity and then take home.

METHODS

Settings and Participants

The model TEM was used in a module as part of a weeklong summer camp for high school juniors (31%) and seniors (69%) at the University of Minnesota. The 2 h activity was conducted with two groups of camp students (n = 25 in week one, n = 22 in week two), including 57% females and 60% students of color. The students that were admitted to the program from the applicant pool demonstrated strong academic performance (69% with a 3.8 GPA or higher on a 4-point scale) and have a background in STEM education (92% have taken chemistry, 72% have taken some college-level STEM coursework, and 95% have participated in at least one STEM-related extracurricular). This study met the criteria for Institutional Review Board exemption.

Building of a Model TEM

A model TEM device (Figure 1) was built with the following goals in mind: (a) the device should result in 2D images, (b) prints analogous to micrographs should be physical and annotatable, (c) the device should have a "mystery" aspect (samples should be able to be kept from view), (d) the device should be capable of achieving a tilt series, and (e) supplies needed to build the device should be easy to obtain for relatively low cost. Our device uses PVC to create the body of the microscope, a high UV output bulb to represent the electron beam, and cyanotyping paper for imaging. Finally, we included a stage that can be both lifted off the base and rotated. The total cost for all materials was approximately $50 USD; build details, including supply lists and diagrammed build steps, can be found in the Supporting Information.

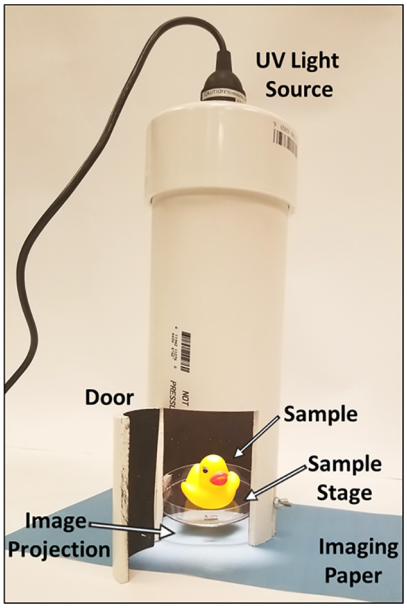

Figure 1. One of the model TEM devices built, showing relevant parts for operation.

PROGRAM DESIGN

Students worked in groups of 5–7 through four activities, each of which involved producing cyanotype prints using the model TEMs. First, students used the "microscope" to create prints of mystery objects (e.g., paper clip, glass marble, 20-sided die, bottle cap) and analyzed prints to identify the objects (Figure 2). Some objects were readily identifiable (e.g., paper clip, tack), some objects were recognizable with mild confidence but could be other things (e.g., quarter, bottle cap), some objects' distinguishability was based on their orientation on the stage (e.g., wing nut, screw), and some objects could not be

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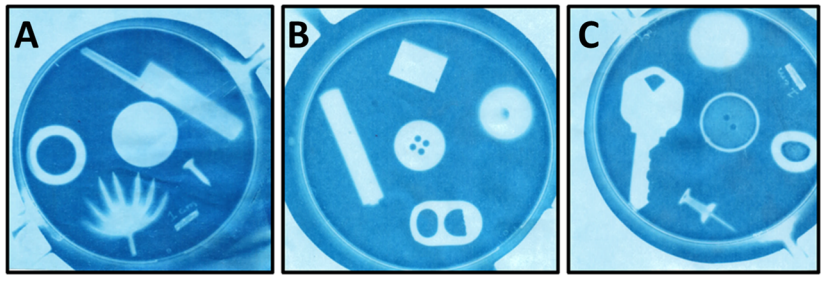

Figure 2. Three cyanotypes of mystery objects prepared for students. Objects imaged are (A) pen cap, quarter, plastic loop ring, screw, plastic aquarium leaf, (B) block of staples, "atom" from chemistry modeling kit, button, pop tab, and AAAA battery, (C) a 20-sided die, glass flat-bottomed marble, button, thumb tack, key.

identified with the information available (e.g., block of staples, glass marble). Students engaged in their groups to discuss object identities and were able to identify many of the objects with relative ease. The most difficult object to identify was the decorative glass marble, which demonstrates pseudodiffraction contrast similar to what is seen in an actual TEM. (see Figure 2, panel C). Variations on the mystery item activity, showing imaging artifacts and the role of sample orientation are shown in Figure S1.

Second, students were introduced to the idea that scientists use TEM micrographs to determine the size of objects such as nanoparticles by performing a coin-measuring activity (Figure 3). Mystery items were switched out for sets of 6−7 coins from

Figure 3. A cyanotype of assorted U.S. currency taken for the sizing activity. Students numbered each coin image and measured diameters.

U.S. currency, which were imaged along with a 1 cm scale bar drawn on the sample stage in permanent marker (seen in Figures 2, 3, and 4). After taking their prints, students were provided with rulers and a table of official diameters of U.S. currency. Groups recorded measurements and determined how many cents were on their stage. Students were able to identify the monetary value of their samples with this method and relate it to sizing processes used by researchers as well as understand the function of scale bars in micrographs.

Third, students used the model TEMs to demonstrate a tilt series. Each group was assigned a different mystery shape which had been molded out of oven-baked clay by one of the researchers. Cyanotype "micrographs" were taken after lifting and tilting the stage. Each group collected three images of their mystery shape: 0 degrees, 30−45 deg, and 90 deg (Figure 4). After collecting all three tilt images, students consulted their "micrographs" to determine and sketch the 3D shape of their objects. Students were generally able to identify the shape and verbally indicated that the addition of the third tilt (90 deg) was particularly helpful. A representative from each group drew the consensus design on the board and then revealed the mystery item to the group.

Finally, students were given sets of various objects (e.g., scrapbooking supplies, small office supplies, pasta, beads, buttons, cut out transparencies, small toys) and were instructed to choose objects to "image" and predict how they would look in their resulting "micrographs" (Figure 5). Student buy-in for this activity was high and many groups made three or more prints.

HAZARDS AND SAFETY PRECAUTIONS

This demonstration uses high-output UV-light bulbs designed for reptile husbandry. Long-term exposure to UV-light can be harmful. We advise that the light bulbs be kept in the PVC column and imaging performed with the door closed to prevent students from looking at this light source directly throughout the course of the activity. UV output and

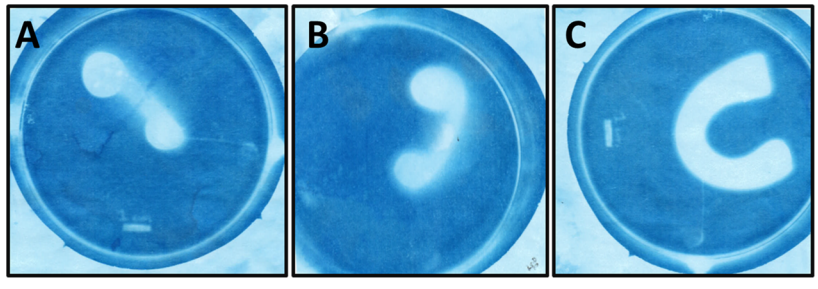

Figure 4. A tilt series of a rainbow-shaped object taken by one of the student groups: (A) 0° degree rotation, (B) approximately 45° degree rotation, (C) 90° degree rotation. (In this particular series, the object was laid flat to achieve a 90° rotation in order to keep activities moving. However, rotation of the stage 90° and the sample simultaneously is achievable by adhering the sample with sufficiently strong double-sided tape.)

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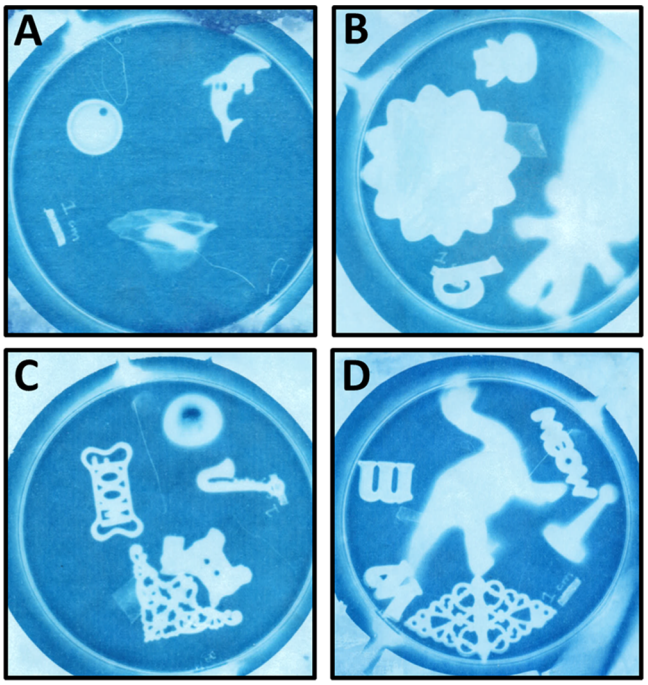

Figure 5. Cyanotypes taken by students freely in the "Predict your Print!" activity. Identities of items listed here where they can be identified. (A) dolphin-shaped scrapbooking brad, bead, double stick tape crumped up. (B) troll doll, flower-shaped decorative pin, penguin-shaped eraser, letter "b", (C) saxophone-shaped bead, marble, dog-shaped bead, scrapbooking page corner, "woof" embellishment. (D) toy dinosaur, embellishment, letter "m", letter "w", board game player marker, "meow" embellishment.

penetration at varying distance data can be obtained from the manufacturer, Exo-Terra.

IMPLEMENTATION

The students' understanding of TEM was assessed with two-tiered surveys administered before the module, after an introductory lecture, and then again after completing the hands-on activities. The short lecture showed students traditional optical microscopy, electron microscopy (TEM, SEM), and images of instrumentation. Students actively participated in the lecture, providing compare/contrast statements for microscopy and offering questions and definitions for terminology. Each survey packet was labeled with a unique number string to allow for individual progress to be tracked while maintaining student anonymity. Surveys included multiple-choice content questions and true/false statements. Each question or statement was then followed by a question for students to rate their confidence in their answer (see Supporting Information, Student Pre-assessment 1, 2, and Post-assessment). The confidence questions asked students to rate their confidence in each answer from 0%−100% in intervals of 20%. During the post-assessment, students were also asked to rate on a 5-point Likert scale how much they had learned and how much they enjoyed the lecture and hands-on portions of the module. The student pre-assessment included only questions 1−6, student post-assessment 1 included questions 1−15, and student post-assessment 2 included questions 1−15 and an additional four (questions 16−19) opinion questions.

To assess the impact of the module on the student understanding and confidence, the three assessments were analyzed for differences. To assess student learning, a X^2 test was done on questions 1−15. For questions 1−6, two X^2 tests were performed—one to compare prelecture to post-lecture and one to compare post-lecture to post-activity. Confidence questions were translated from a scale (0%−20% to 80%−100% confident) to an ordinal scale (1 to 5) for analysis in a paired *t*-test.

The 5-point Likert scale questions on the post-activity assessment asked students to indicate how much they agreed with statements about how much they learned and how much they enjoyed the lecture and hands-on portions of the module.

CONCLUSION

For this student group, significant learning gains were shown for question 5 and question 13 after the model TEM hands-on activities (Figure 6). Question 5 relates only to the resolution of TEM and therefore should not be influenced by the activity, only by repetition effects. Question 13 relates to interpretation of a tilt series. The difference in correctness between post-lecture and post-activity on question 13 indicates that this activity demonstrates the effect of a tilt series well and may aid students in developing the spatial reasoning skills to understand this method of characterization.

Despite few statistically significant gains in student knowledge, there were many significant gains in student confidence between post-lecture and post-activity surveys (questions 2, 7, 8, 10, 11, 12, 13, 14, 15) (Figure 6 and Table S1). Research on student confidence indicates that student confidence is generally correlated to student knowledge.[20] On the basis of gains in student confidence, we conclude that students were guessing less on the listed questions for the post-activity survey

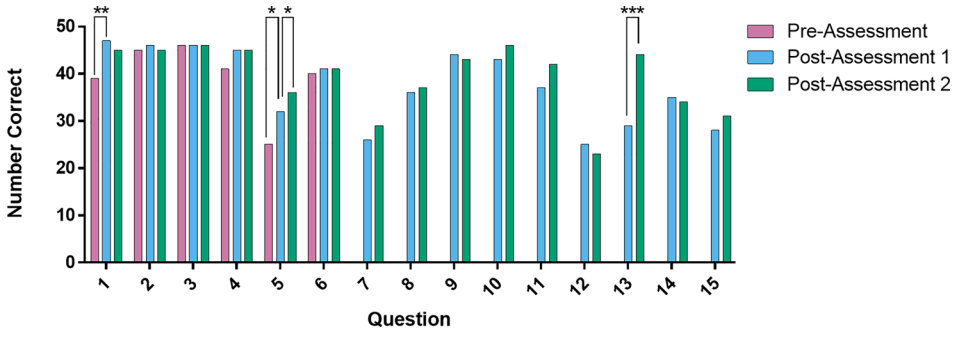

Figure 6. Number of students answering each question correctly. $N = 47$. * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$, as determined by X^2 test. The dashed line visually separates questions surveyed thrice (1−6) and those surveyed twice (7−15).

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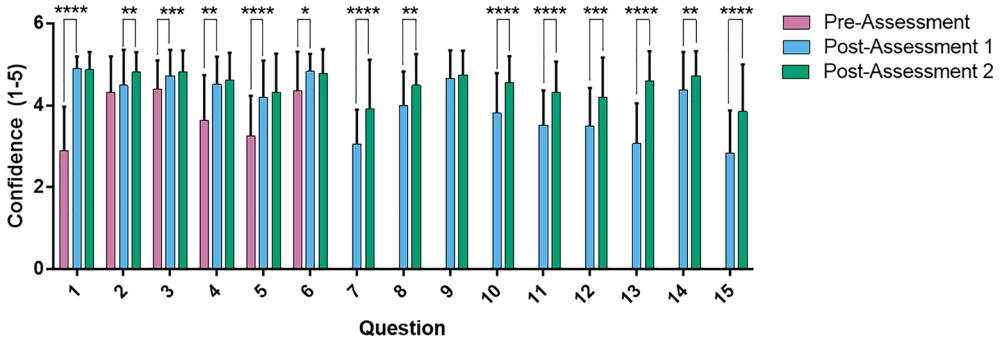

Figure 7. Student confidence on each question at pre-1, pre-2 or post-activity survey times. $N = 47$. * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$, **** $p < 0.0001$ as determined by paired t-test. Dashed line visually separates questions surveyed thrice (1−6) and those surveyed twice (7−15).

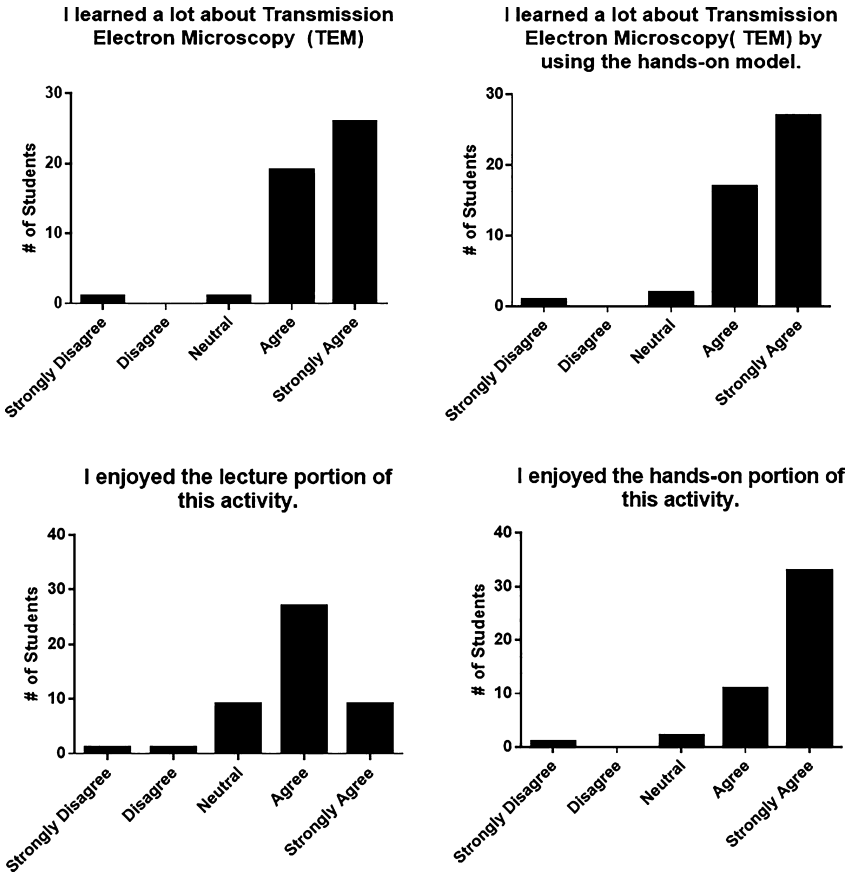

Figure 8. Student responses to statements regarding their view on educational value and enjoyment of the lecture and hands-on portions. Scale converted to numerical (strongly agree = 5, etc.) to report average response.

and might have had individual, but not statistically significant learning gains as a cohort. This cohort is considered both science-attentive and high-performing, and therefore, we suggest that the difference in metacognitive judgment (confidence) indicates learning gains that would otherwise be masked by strong test-taking skills. Question 13 showed both learning and confidence gains (Figures 6 and 7 and Table S2), indicating that the hands-on tilt-series activity was useful for students to connect consecutive, changing sample orientations to results on "micrographs." In both sessions, all groups (8 total) were able to correctly draw their mystery sample shape after collecting three angles in a tilt series, offering anecdotal corroboration for the survey data. Finally, while students did not self-report any learning differences between the lecture and the hands-on portions (Figure 8), they indicated that they enjoyed the hands-on portion of the lesson much more than the lecture. As student enjoyment is predictive of further engagement in science topics, the fact that students found this activity more enjoyable than a traditional lecture suggests that it has value in promoting engagement and future science learning.[21]

Following the successes with this group of students, future work will present this activity to different cohorts of students with varied educational background and level. While this group had high buy-in for the activity and a generally positive response to the hands-on portion, this lesson was presented in a "capsule" format, and it will be interesting to explore how this activity can fit into a larger course context. This module may also be expanded further by pairing it with analysis of real TEM micrographs, remote access programs, or TEM access as

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well as the potential for students to further explore the concepts of cyanotyping. Suggestions for additional curriculum with the model can be found in the Supporting Information.

Overall, through use of this simple model of a transmission electron microscope, students demonstrated (i) use of strengths and limitations of their characterization technique in order to identify mystery items, (ii) use of image scale bars as applied to sizing "particles" (U.S. coinage) (iii) use of 2D plan view "micrograph" series to identify a 3D object's general shape, and (iv) creative expression in the "Predict your Print!" portion.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.8b01059.

Instructions for building the model, tips and recommendations for operation, surveys used for assessment, and worksheets provided to students throughout the activities (PDF)

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Notes

The authors declare no competing financial interest.

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