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Laboratory Experiment

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Fabrication of Polyvinylpyrrolidone Micro-/Nanostructures Utilizing Microcontact Printing

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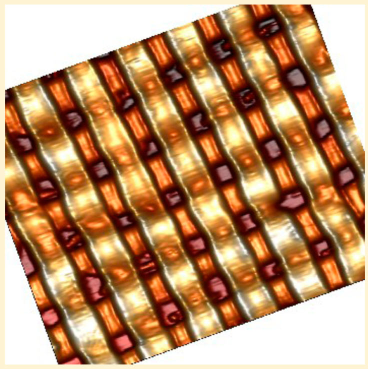
Ⓢ *Supporting Information*

ABSTRACT: This paper describes a laboratory exercise that provides students enrolled in introductory nanotechnology courses with an opportunity to synthesize polymer structures with micro- and nanoscale dimensions. Polyvinylpyrrolidone (PVP) films deposited on corrugated PDMS stamps using student-built spin coaters were transferred to clean, dry substrates via microcontact printing. The microscale dimensions of the resulting patterns were characterized in class using optical microscopy. Characterization with atomic force microscopy (AFM) was used for visualization of nanoscale vertical dimensions of the structures. This laboratory investigation highlights the following concepts often associated with polymer nanostructure fabrication: polymer synthesis, surface chemistry, soft lithography, and contact angle. It is noteworthy to mention that this laboratory exercise demonstrates the feasibility of utilizing nontoxic, cost-effective, bench-top materials to teach and investigate fundamentals associated with fabrication of polymer nanomaterials.



KEYWORDS: *Analytical Chemistry, Materials Science, Laboratory Instruction, Hands-On Learning/Manipulatives, Nanotechnology, Surface Science, First-Year Undergraduate/General, Polymer Chemistry*

■ INTRODUCTION

Polymers and thin films are of increasing technological value to society, particularly in the medical and electronic fields.[1] Polymer structures can be used as scaffolds, barriers, and for drug delivery systems.[2] In the electronics field, polymer structures have been used to create insulators and semiconducting structures in thin film transistor arrays.[3,4] Due to the increasing appearance of nanoscale polymer structures in a variety of medical and electronic applications, development of inexpensive and creative methods that teach the fundamental behavior and fabrication of these structures is necessary.[1] Historically, polymer nanostructure fabrication and utility has not been extensively addressed in undergraduate curriculum, which underscores the need for laboratory modules that introduce associated content.[1] There are reports that describe non-cleanroom based nanofabrication laboratory exercises that address this issue.[1,5−10] However, the laboratory exercise reported in this manuscript is unique because it provides students with an opportunity to participate in polymer nanofabrication at community colleges and undergraduate institutions that may not have access to specialized equipment. This is accomplished by eliminating the necessity of unique substrate pretreatment procedures. Reports state that, oftentimes, polymer nanomaterial synthesis requires special procedures, such as plasma treatment,[5] ozone patterning,[1] alkanethiol deposition,[11] etching,[6] electrochemical deposition,[7] and application of heat,[8] prior to deposition of polymer nanostructures.

Soft Lithography

The nanofabrication method that is used in this investigation is soft lithography, which is a form of replica molding for fabricating micro- and nanoscale structures. It utilizes an elastomeric stamp with patterned relief structures on its surface to create nanomaterials with feature sizes ranging from 30 nm to 100 μm.[12] Elastomers are used in this process because they can make conformal contact with substrates.[12] Soft lithography encompasses a family of techniques based on the process of molding a soft polymer on hard masters; the resulting elastomeric molds are used in conjunction with a molecular "ink" to control specific properties of surfaces at the micro- and nanoscale levels.[12] Elastomeric stamps are commonly made from polydimethylsiloxane (PDMS).[13] PDMS is an elastomeric stamp that has been used in laboratory exercises for creation of superhydrophobic surfaces[5] and of nanocrystalline patterns,[14] printing of alkanethiols,[6] deposition of polycarbonate,[8] and deposition of nanoparticle infused samples.[9]

Microcontact printing is the specific form of soft lithography used in this laboratory exercise. This technique involves the use of protrusions on the surface of an elastomeric stamp inked with molecules to pattern structures on flat substrates.[15] Microcontact printing of polymer nanostructures has involved carbon nanotube polymer multilevel films for organic thin film transistors,[16] micellar thin films,[17] print patterns for electroluminescent displays,[11] OLEDs,[18] and for the preparation of optical waveguides.[19] Additionally, microcontact printing has been used to create patterns to study cell-surface interactions,[20]

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J. Chem. Educ. XXXX, XXX, XXX−XXX

micropatterning of conducting polymer layers,[21] and patterns of organic thin film transistors.[22] This laboratory investigation employs the novel use of poly(vinylpyrrolidone) (PVP) (Figure 1), a safe, nontoxic polymer that readily forms films and structures.[2] There are no reports that describe the fabrication of PVP structures or deposition of PVP films using microcontact printing. PVP is extremely soluble in water and exhibits strong hydrogen bonding; these characteristics are believed to be the reasons for the ability of PVP to readily form thin films.[2]

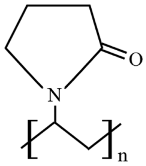


Figure 1. Structure of PVP.

■ EXPERIMENT

Instructor Preparations

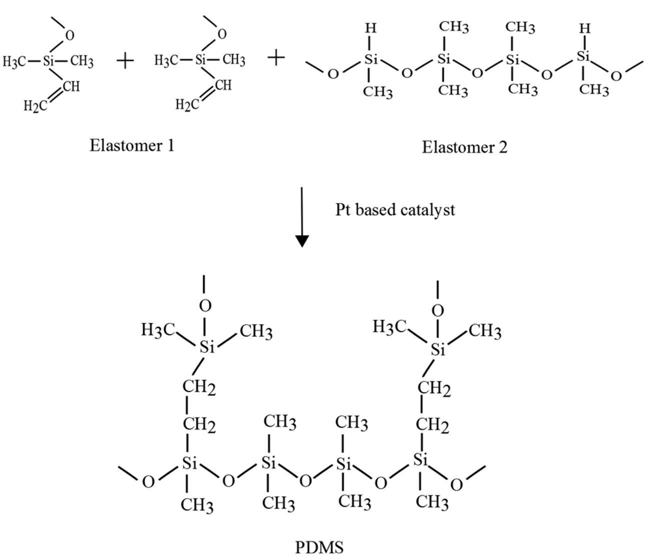
PDMS stamps were prepared using commercially available recordable compact discs (CD-Rs) according to the procedures listed in the Supporting Information. Faculty prepared a 0.2% (w/v) PVP solution using 100% isopropyl alcohol prior to the start of the laboratory exercise.

Prelab Instruction

Before facilitating the experiment, faculty presented a prelab lecture describing the basic concepts associated with the laboratory experiment. This section included a description of PDMS, stating that it is made from a special type of polymer known as an elastomer. Furthermore, a brief overview of elastomers was provided, describing them as large molecules with little rigidity and very weak intermolecular interactions.[23] It is also stated that elastomeric molecules are encouraged to entangle through the use of a cross-linking agent[9,13](Scheme 1).

During the prelab lecture, single drops of water and isopropyl alcohol were placed on the surface of two separate PDMS samples as shown in Figure 2. Water was observed beading up and the alcohol spreading out on separate PDMS surfaces.

Scheme 1. Synthesis of PDMS



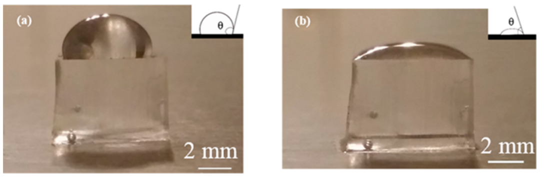


Figure 2. Drop of water on the surface of PDMS (a). Inset illustrates a contact angle (θ) greater than 90°. Drop of isopropyl alcohol on the surface of PDMS (b). Inset illustrates a contact angle (θ) less than 90°.

Students were informed that water beads up on the surface of PDMS, because PDMS is nonpolar and therefore hydrophobic. Justification of this observation was provided by mentioning that water is a polar substance which means there is greater attraction between water molecules within the drop of water than between water molecules and PDMS. However, isopropyl alcohol has polar and nonpolar characteristics. The carbon chain in the structure of isopropyl alcohol allows it to sufficiently wet the surface of the PDMS stamp. In this demonstration, contact angle is mentioned, which is described as a measurement often used to express the fundamental surface properties of materials,[24] particularly the wetting properties of a liquid/solid system.[25] Contact angle is the angle formed between the solid surface and a line tangent to the surface of the drop at the point of contact of the two lines.[24] The magnitude of the contact angle depends on the balance between the adhesive forces between the liquid and the solid surface and the cohesive forces within the liquid that counteract spreading.[24] A contact angle greater than 90° indicates that cohesive forces within the liquid dominate and the liquid will not spread on the solid surface, and a contact angle less than 90° indicates that adhesive forces dominate and a liquid does wet the solid surface.[24] Students are made aware of the fact that although PVP is very soluble in water, an aqueous PVP solution is not the optimal ink to use because students observe that water does not sufficiently wet the surface of the patterned PDMS to create a film for transfer.

On the basis of reports describing the behavior of PVP in solutions,[26−28] it is believed that carbonyl groups of PVP (C=O) form hydrogen bonds with hydrogen atoms of hydroxyl groups (O−H) found in isopropyl alcohol (Figure 3). For this reason, and due to the ability of isopropyl alcohol to sufficiently wet the surface of PDMS, PVP solutions used for pattern transfer were prepared using isopropyl alcohol.

Faculty demonstrated the use of AFM for pattern characterization at the end of the laboratory exercise. Incorporation of a characterization component using specialized forms of

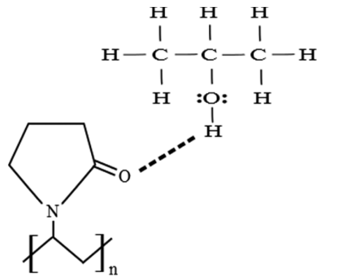


Figure 3. Hydrogen bonding between PVP and isopropyl alcohol.

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J. Chem. Educ. XXXX, XXX, XXX−XXX

microscopy is consistent with existing literature precedents that describe the use of scanning probe microscopes in undergraduate experiments.[29−44] A previous lecture was used to describe AFM operation. An explanation of the laser detection system, and how it monitors the vertical movements of an AFM tip scanning a sample surface, was included. In this lecture, students learn that an AFM tip is mounted at the end of a flexible cantilever that bends in response to tip movement. The extent of bending is determined using a laser beam that is reflected off the back of a cantilever, and the reflected beam strikes a photodetector, which is a four quadrant light sensor used to produce signals proportional to the heights of surface features. It is mentioned that signals generated by the photodetector are used to prepare topographical maps of nano- and microscale surface features[43,44] (Figure 4).

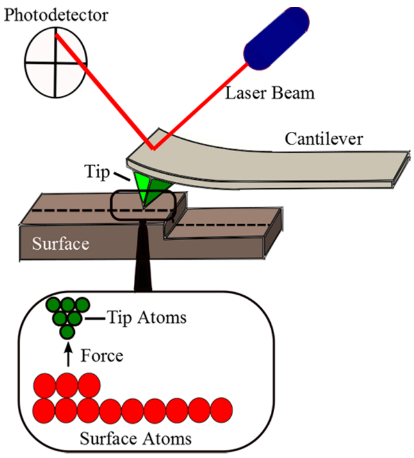

Figure 4. Diagram illustrating AFM operation.

Microcontact Printing

The concept of microcontact printing was introduced during the prelab lecture. It was shown that microcontact printing involves using flexible PDMS stamps (Figure 5a) "inked" with material to be transferred (Figure 5b). It is important to note that the "inking" step in the laboratory exercise described in this manuscript involves the use of a student-built spin coater to uniformly deposit a patterned PVP film for transfer. Once properly inked, the PDMS stamp is placed on a flat substrate (Figure 5c). The stamp is then removed and the pattern remains on the substrate (Figure 5d).

Execution

This laboratory experiment was used in an introductory nanotechnology course. Implementation involved a 25 min prelab lecture, which included demonstrating the assembly of the spin coater (Figure 6). Use of spin coating was a necessity; it provided a means to uniformly deposit a PVP film on the surface of a patterned PDMS stamp for transfer. Participating students worked in small groups (3−4 students) taking approximately 30 min to complete the laboratory experiment. Following pattern deposition and optical characterization, an AFM analysis was used to assess the dimensions of the patterns. AFM characterization lasted approximately 25 min. At the completion of the laboratory experiment, assessment of student understanding of associated concepts was achieved using online quizzes prepared by faculty.

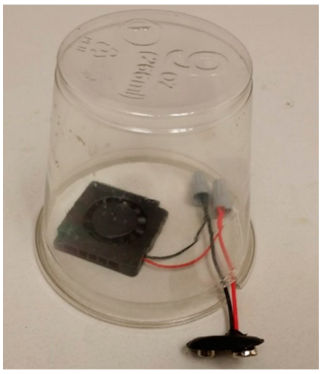

Figure 6. Home-built spin coater.

■ HAZARDS

Isopropyl alcohol is flammable; do not use alcohol near an open flame or heat source.

■ RESULTS

Upon successfully depositing the patterned polymer film, students observed an optically transparent polymer film (Figure 7a) that showed strong evidence of a prismatic effect (Figure 7b).

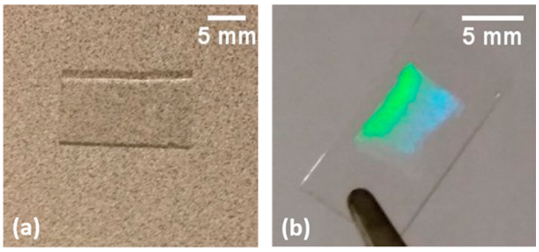

Figure 7. Transparency of deposited PVP film (a). Prismatic effect observed after stamping PVP pattern (b).

Optical microscopy

Toward the end of the microcontact printing procedure, students used a Swift M3200 Optical Microscope, with no filters and operating in a bright-field configuration, to view the

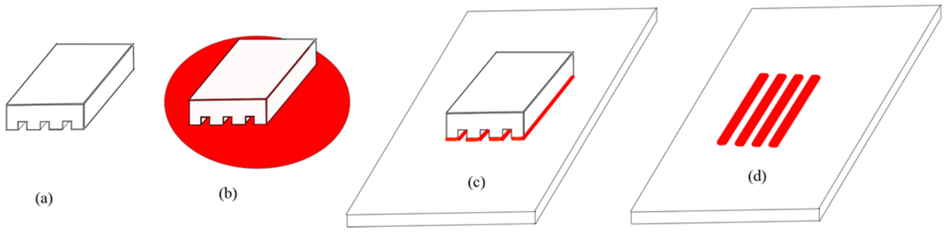

Figure 5. Diagram illustrating microcontact printing steps.

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J. Chem. Educ. XXXX, XXX, XXX−XXX

microscale dimensions of the polymer structures. A 10× ocular lens and a 10× objective lens was used to optically characterize the structures (Figure 8).

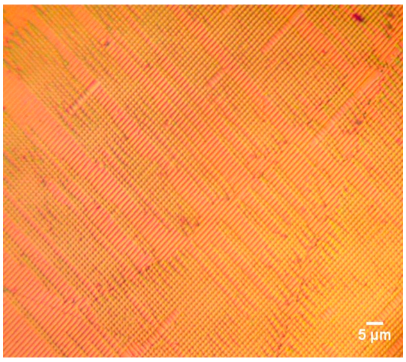

Figure 8. Optical images of PVP patterns magnified at 100×.

Atomic Force Microscopy

During the fall 2014 semester, faculty used an Agilent 5400 AFM outside of class to generate images (Figure 9) to share with students during subsequent lectures. The following spring 2015 semester, faculty demonstrated characterization of the student-generated polymer structures in class using a portable Nanosurf NaioAFM System (Nanoscience Instruments, Inc.) WSXM image processing software[45] was used to create a cross-sectional profile (Figure 10, left) of the AFM data (Figure 10, right) to provide dimensional information regarding the polymer structures. The profile indicates that the polymer structures students made were approximately 1 μm wide and 35 nm tall. During the AFM characterization portion of the experiment, it was mentioned that the lateral microscale widths of the structures makes it possible to characterize the patterns optically; however, nanoscale thicknesses of the structures had to be assessed using AFM.

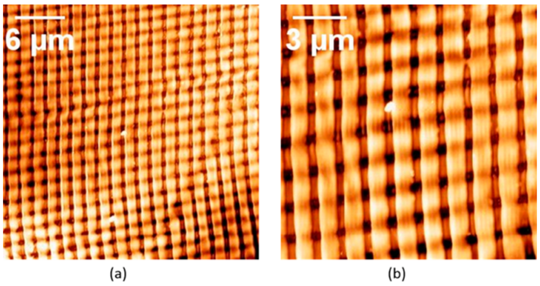

Figure 9. The 30 μm × 30 μm (a) and 15 μm × 15 μm (b) scans of PVP structures.

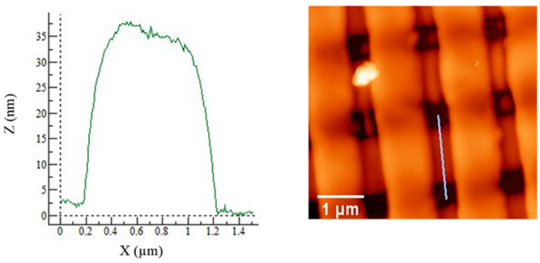

Figure 10. Cross-section plot (left) obtained from an AFM scan of PVP structures (right).

■ SUMMARY

This laboratory investigation was successfully implemented for the first time in introductory nanotechnology classes during the fall 2014 semester, and again in the spring 2015 semester. This exercise allowed students to fabricate polymer structures with microscale and nanoscale dimensions. This laboratory exercises also allows students to characterize the structures with an optical microscope and AFM to assess the dimensions of the polymer structures they make in class. Understanding of the fundamental science and processes involved with this laboratory experiment was measured via postlab quiz questions, included in the Supporting Information section. Average postlab quiz results of 90% suggest that students grasped rudimentary concepts associated with polymer nanostructure fabrication and subsequent AFM characterization.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Instructor notes are available that include a parts list for the student-built spin coater, assembly of the spin coater, procedures for PDMS stamp preparation, determination of the PVP solution to use for optimal film production, and post-lab quiz questions. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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