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3D Printed Block Copolymer Nanostructures

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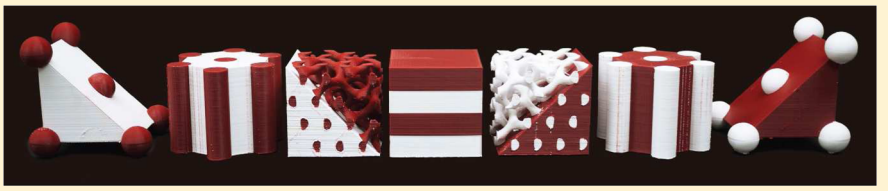
Supporting Information



ABSTRACT: The emergence of 3D printing has dramatically advanced the availability of tangible molecular and extended solid models. Interestingly, there are few nanostructure models available both commercially and through other do-it-yourself approaches such as 3D printing. This is unfortunate given the importance of nanotechnology in science today. In this work, we have filled part of this gap by designing and 3D printing several block copolymer (BCP) nanostructure morphologies. We used a variety of methods including manually drawing the files within 3D computer design software, using equations with mathematical graphing software, and developing a programming script to convert self-consistent field theory (SCFT) structure data into a 3D printable file. Conversion of SCF data into 3D printable structures may find broader applicability beyond creating BCP nanostructures as SCF calculations are used in a variety of geometric computations. All methods reported herein produced tangible 3D prints of approximately equal quality. These tangible models will be useful for educators, students, and researchers in polymer science and nanotechnology.

KEYWORDS: *General Public, Graduate Education/Research, Polymer Chemistry, Hands-On Learning/Manipulatives, Crystals/Crystallography, Nanotechnology*

INTRODUCTION

Advances in nanotechnology are impacting every field of science. As such, nanotechnology topics are increasingly being taught at the K−12 and introductory undergraduate level, either integrated in subject specific courses or as stand-alone topics.[1−8] Since the properties of nanoscale objects are strongly influenced by their structure, it is important for students to have a good appreciation and understanding of the structures themselves. However, this can be challenging because of the often intricate shape of nanoparticles or complex periodic domains of nanostructures.

The chemical sciences have a strong tradition of visualizing and representing structures of molecules and extended solids using tangible models.[9−16] These tangible models are especially valuable in chemical education as they help students understand the spatial arrangement, bonding, and symmetry of molecules. In the same way in which physical molecular models are indispensable learning aids in the chemical sciences, nanotechnology education can greatly benefit from the use of tangible 3D models of nanostructures.[4,6,17]

3D printing is an additive manufacturing process that is capable of producing complex models.[18,19] Recently, 3D printing has been used in our own research[20] as well as by other researchers[21−27] as a powerful technique to create complex representations of chemical structures directly from X-ray crystallography data, overcoming many conventional limitations of commercial molecular model kits. The ability of 3D printers to create arbitrarily complex shapes from digital files makes it an ideal technique for constructing models of nanostructured materials.

As a demonstration to the power of 3D printing in visualizing nanostructured objects, we have chosen to depict the diverse and often complex nanostructures that form during the self-assembly of AB diblock copolymers including spheres on a body-centered cube (S_{BCC}), hexagonally packed cylinders (H), gyroid (G), and lamellae (L) (Figure 1). Block copolymers (BCPs) are macromolecules composed of two or more distinct polymer chains that are covalently connected. BCPs can spontaneously undergo microphase separation to form self-assembled nanostructured periodic domains on the length scale of 10−100 nm.[28−31] This self-assembly is driven by the inherent immiscibility of the chemically distinct polymers. The resulting morphology is determined by the chemical identity of the polymer blocks, molecular weight, the connectivity architecture, and finally the processing conditions. More intricate morphologies besides the classic S_{BCC}, H, G, and L morphologies can be obtained with additional polymer segments or by forming composite materials. For example, the addition of just one polymer block to form an ABC linear triblock copolymer can result in further complexities and a wide variety of exquisite periodic nanostructures such as cylinders at

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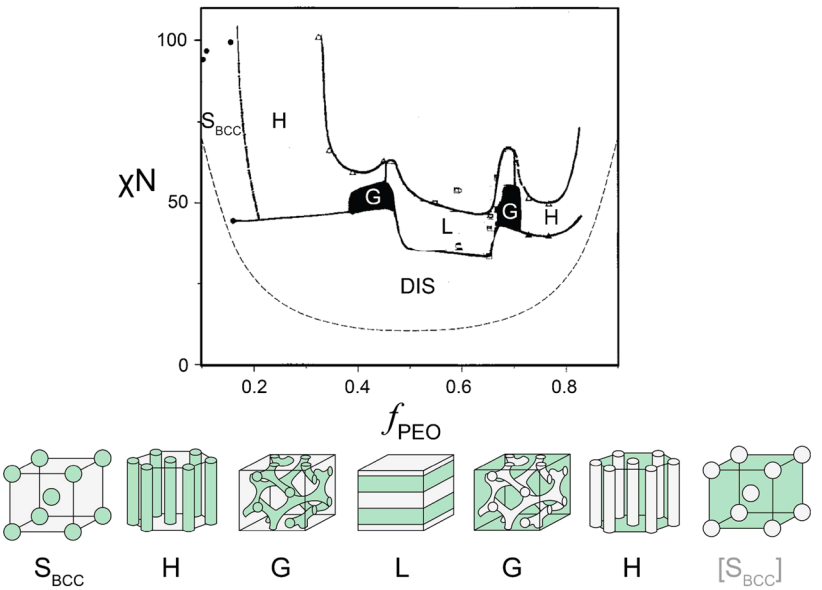

Figure 1. Experimental phase diagram for an AB diblock copolymer, polyisoprene-*b*-poly(ethylene oxide) (PI−PEO) depicting several equilibrium morphologies including spheres on a body-centered cube (S_{BCC}), hexagonally packed cylinders (H), gyroid (G), lamellae (L), and a homogeneous disordered (DIS) state. Data markers represent the PI−PEO block copolymer samples; solid lines represent phase boundaries. The *y*-axis plots the incompatibility of the polymer blocks as the product χN. χ is the Flory−Huggins interaction parameter, and N is a segmental volume. The *x*-axis, f_{PEO}, is the volume fraction of the PEO block. If the product χN is large enough, the PI−PEO block copolymer will no longer be disordered and will phase separate into an ordered morphology (either S_{BCC}, H, G, or L). Adjustments to the PEO volume fraction results in different equilibrium morphologies. Moreover, there are multiple order-to-order morphology phase transitions that can occur in a single BCP sample simply by changing χN (e.g., changes in temperature). Figure adapted with permission from ref 41. Copyright 2001 American Chemical Society.

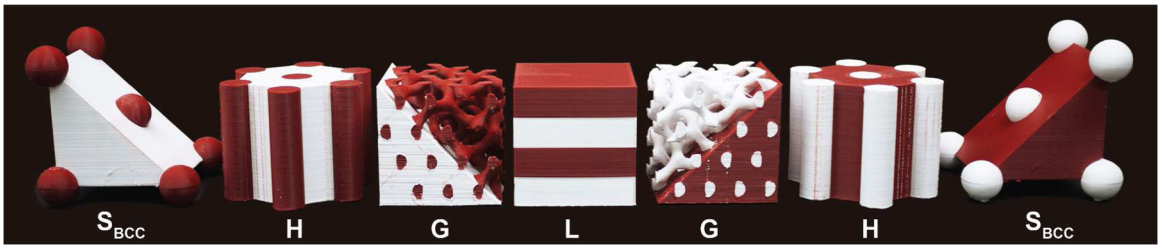

Figure 2. 3D printed AB diblock copolymer morphologies including spheres on a body-centered cube (S_{BCC}), hexagonally packed cylinders (H), gyroid (G), and lamellae (L). Structures include both sides of the phase diagram, where to the left of the L morphology, the majority component is block A (white) and to the right of the L morphology, the majority component is block B (red). The structures were 3D printed in ABS plastic with approximate dimensions of 8 cm in all directions.

a lamellar interface,[32,33] knitting pattern,[33−35] spheres on spheres,[36] spheres on cylinders,[37] and many more.[38−40]

Given the wide range of BCP-based nanotechnology applications,[42] combined with the feasibility of synthesizing block copolymers in undergraduate laboratories,[43−46] BCP morphologies are an excellent test case for illustrating the capabilities of 3D printing to visualize complex nanostructures. In this work we 3D printed the nanostructures formed through the self-assembly of AB diblock copolymers as well as two example linear ABC triblock morphologies. All of the 3D digital files have been included as Supporting Information for those interested in reproducing our results.

GENERAL METHODOLOGY

Detailed specifications of software, equipment, and methods are available in the Supporting Information. In brief, the majority of BCP 3D digital models were drawn in 3D design software including spheres on a body-centered cube, hexagonally packed cylinders, lamellae, cylinders at a lamellar interface, and the knitting pattern. The gyroid 3D structure was modeled in a mathematical visualization program. Moreover, an additional gyroid 3D structure was produced by converting SCFT data into a 3D surface using a custom programming script. All digital 3D BCP models were ultimately converted into a .stl file for 3D printing on a MakerBot Replicator 2X 3D printer equipped with ABS feed material.

RESULTS AND DISCUSSION

We successfully 3D printed the AB diblock copolymer self-assembled nanostructures including the morphologies spheres on S_{BCC}, H, G, and L. Two linear ABC block copolymer morphologies including the cylinders at the lamellar interface and the knitting pattern were also printed (Figures 2 and 3). Notably, for many of the structures we opted to design and include more of the structure than what would appear in a single crystallographic unit cell. All structures printed with approximately equal quality and with good mechanical integrity. To represent the unique A and B polymer segments within the single component (covalently joined) AB diblock copolymer system, we used different colors of ABS plastic for the A and B

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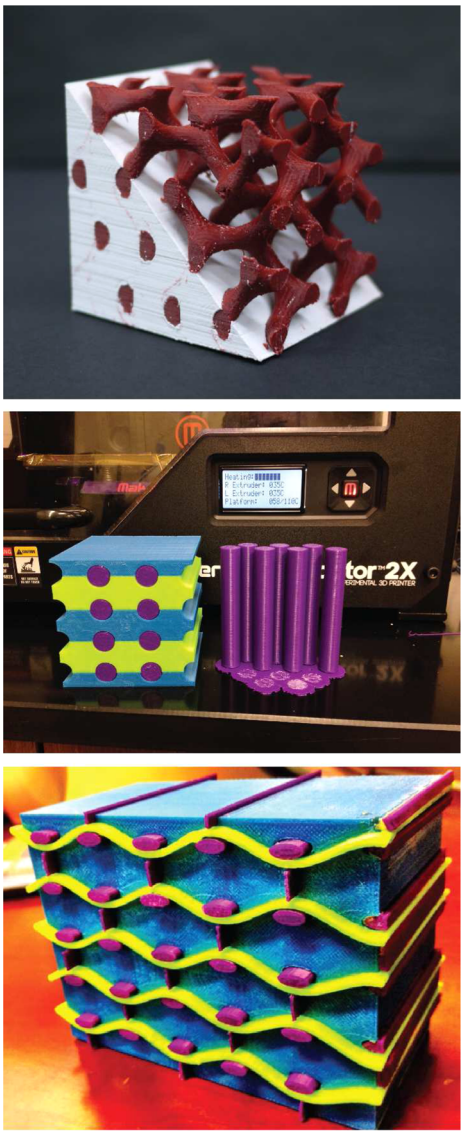

Figure 3. Close-up photograph of the 3D printed AB diblock gyroid (G) morphology (top). 3D printed ABC triblock copolymer self-assembled morphology and B cylinders at A/C lamellar interface (middle), and the ABC triblock copolymer knitting pattern (bottom). The structures were 3D printed in ABS plastic with approximate dimensions of 8 cm in all directions.

segments. This allowed both sides of the AB diblock copolymer phase diagram to be easily discernible. For example, in Figure 2 (left S_{BCC} structure), the minority component A, red, forms the spheres, while the majority component B, white, encompasses the matrix around the spheres. Conversely, on the opposite side of the phase diagram (right S_{BCC} structure), the minority component is now the B block in white, which forms the minority spherical component of the structure, while the majority component is the A block in red. Similarly, the negative image counterparts of the H and G structures were also created to represent the change in volume fraction (and morphology) when horizontally traversing one side of the phase diagram to the other.

Digital structure and 3D file representations of nanostructures such as the BCP morphologies in this work are not readily available. This is in direct contrast with molecular and extended solid structures for which there are hundreds of thousands of crystallographic information files (.cif) contained in repositories such as the Cambridge Structural Database, Inorganic Crystal Structure Database, and Crystallography Open Database.[47] With the appropriate software packages, these molecular and extended solid .cif files can be easily converted into 3D representations that are suitable for 3D printing.[20−22,24,26] While many molecular and extended solid structures share the same symmetry and space groups as the BCP morphologies, they fail at representing the polymeric domains and surface topology. Therefore, we had to design our own representations in 3D design software to depict the unique domains and surfaces of the self-assembled BCPs (Supporting Information).

We realized, however, that there is a tremendous opportunity to overcome this limitation given the large amount of successful research using advanced calculations to model and theoretically predict BCP morphologies (e.g., self-consistent field theory, SCFT).[31] The limitation of having to design the 3D structures with traditional 3D design software is eliminated with such calculations. As a proof of concept, we successfully converted SCF data into a 3D model suitable for 3D printing using a programming script that selects and exports all *XYZ* points within a volume fraction of choice (Figure 4). These *XYZ* coordinates were then converted into a printable .stl file (Supporting Information). We suspect this method will find wide utility among theoretical BCP researchers, particularly when investigating difficult to visualize structures and new morphologies. In addition, we anticipate that researchers and educators performing SCF calculations for molecular geometry determinations outside of polymer science may be able to use similar conversion methods to produce 3D models of small molecules, bio-macromolecules, and other structures. Other related computer simulation methods such as cell dynamics simulations[48] can generate similar raw output data, which can be processed into printable 3D models using the exact same techniques presented here.

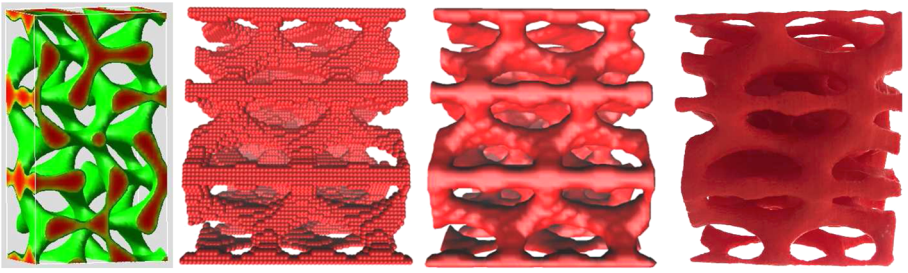

Figure 4. Representations of the gyroid BCP: SCFT data (left); XYZ file visualized in VMD with VDW drawing method and QuickSurf drawing method (middle); and 3D printed structure (right).

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We are currently using these 3D printed models in secondary science outreach activities to teach students about nanostructures. The models were also used in a graduate polymer chemistry course during lectures of polymer characterization techniques to convey the structures of self-assembled BCP morphologies and introduce basic crystallography concepts such as symmetry, spatial arrangement, crystallographic planes, unit cells, and space groups. These models will be particularly useful in the classroom or in research settings when analyzing electron micrographs. In electron micrographs, the appearance of the gyroid morphology (and other morphologies) can differ greatly depending on the unit cell orientation in the field-of-view.[49] With 3D printed nanostructure models in hand, students and researchers can quickly compare the electron micrographs with that of an idealized geometry.

For those looking to reproduce 3D prints of molecular structures or nanostructures such as those within this work, careful consideration should be taken regarding the choice of 3D printer. A full comparison of 3D printers is beyond the scope of this work; however, a few points are worth mentioning. We have logged many hours on both hobbyist level MakerBot melt extrusion 3D printers and professional grade Stratasys 3D printers. In our experiences, both levels of 3D printers can fabricate high quality plastic models if appropriate settings are selected and setup/regular calibration is performed. The major difference is *reliability*. Extruder clogs and part separation from the modeling tray are common in the hobbyist level 3D printers. Several attempts may be necessary to produce an acceptable print. In contrast, the professional grade Stratasys 3D printers we have used are extremely reliable and require little input from the user. We hope that in the near future hobbyist level printer reliability will increase to professional grade levels. Another consideration is if the 3D printer is compatible with a dissolvable support material. Sacrificial support material can be cleanly dissolved away using an appropriate solution (e.g., limonene) that degrades the support polymeric material, while leaving the desired part material completely intact. For example, in 3D printed structures composed of polylactide and ABS, the polylactide is selectively removed with an alkaline solution. This method makes removing support material from complex geometries such as those found in chemical structures straightforward and very efficient. Many 3D printers, including both Stratasys and MakerBots mentioned earlier, can use dissolvable support material.

CONCLUSIONS

Unlike molecular and extended solid chemical structures, there are few periodic nanostructure 3D models available for 3D printing. To overcome this, we have created a series of 3D BCP nanostructure digital models through a variety of methods including manually drawing with 3D computer design applications, using equations with mathematical graphing software, and developing a computer script to convert SCFT data. These conversion methods will be of particular interest to BCP theoretical researchers. All methods in this work produced successful 3D prints of the BCP nanostructures. These nanostructure models should find wide application in nanotechnology teaching and research. We also hope this work will inspire researchers using SCFT for geometry calculations outside of polymer science to consider converting their data into 3D printable structures.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00375.

Detailed methodologies, and programming script to convert BCP SCFT data into 3D printable structures (PDF, DOCX)

3D models (.stl files) of all nanostructures (ZIP)

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Notes

The authors declare no competing financial interest.

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