

iCVD Polymer Microparticle Synthesis Inside a New Liquid Crystal

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Abstract:

Liquid crystals (LCs) can be used as templates to influence polymer morphology during initiated chemical vapor deposition (iCVD). In this study, the liquid crystal TL205 was investigated as a new templating material and compared with the commonly used liquid crystal E7. Polymer was deposited using iCVD under varying deposition parameters, and the resulting morphologies were characterized to determine how polymer growth differed between the two LC templates. Differences in morphology were observed as a function of deposition conditions and LC template, demonstrating that the choice of liquid crystal can influence the resulting polymer structure. To investigate a potential explanation for these differences, the solubility of the monomer in TL205 and E7 was evaluated. The solubility results provide an indication that differences in monomer-LC interactions may contribute to the observed morphological behavior. However, more characterization and experiments are needed to fully determine what is responsible for the differences between TL205 and E7 polymer

molecular interactions, and viscosity can affect polymer nucleation, growth, particle size, and morphology. Therefore, changing the liquid crystal could result in changes to resulting polymer particle size, morphology, etc. [1] [2]

Initiated Chemical Vapor Deposition (iCVD) is a solvent-free technique that can be used to synthesize polymers into the liquid crystal. During iCVD, a monomer, divinylbenzene (DVB) and initiator, tert-butyl peroxide (TBPO), are introduced into the chamber, where a heated filament breaks the initiator, into free radicals that initiate polymerization. Because process parameters such as filament temperature, chamber pressure, flow rates, and deposition time can be controlled independently, iCVD provides a useful method for investigating how different process parameters affect polymer formation. [1] The iCVD reactor setup is shown in figure 1.

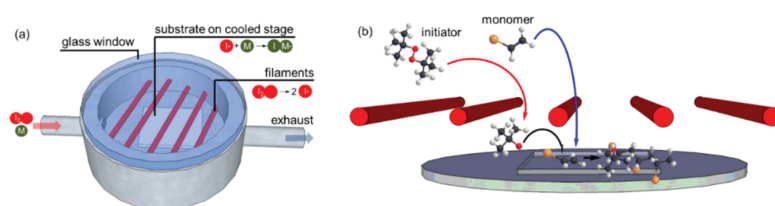


Figure 1: iCVD Reactor Setup.

Summary of Research:

Liquid crystals are materials that have properties of both liquids and crystalline solids. Although they are able to flow like a liquid, their molecules maintain a degree of orientational order. This combination of fluidity and molecular organization makes liquid crystals useful as environments for controlling polymer growth. The molecular organization and chemical properties of a liquid crystal can influence how a polymer forms inside of it. Factors such as monomer solubility,

In this study, a novel and fluorinated liquid crystal, TL205 (TNI=87-90°C), was investigated as a template for polymer growth, and compared to a standard and well investigated liquid crystal, E7 (TNI=60-63). The goal was to determine how the choice of liquid crystal affects the resulting polymer formations. Experiments using a variety of iCVD parameters revealed a difference in

polymer particle size and morphology between the two liquid crystal templates. After establishing that these findings were consistent among a wide range of process parameters, further experiments were conducted to find the reason for these differences. A solubility test was done to determine if the reason for the different polymer particle sizes and morphologies is due to a difference in concentration of monomer in the different liquid crystals. This study provides insight into how different liquid crystals behave as a template for polymer growth during iCVD.

Experiments were completed with varying filament temperature, initiator/monomer flow rate ratio, and deposition time. In one experiment that gave particularly clear results, the filament was heated to $\approx 300^{\circ}\text{C}$, a high initiator/monomer ratio of 0.7 was used, and a featured deposition time of 120 minutes. In figure 2, the resulting polymer formation when an E7 template was used can be seen. The polymer particles, which are roughly $1.7\text{ }\mu\text{m}$ in diameter, are arranged in an energetically favorable hexagonal array. Qualitatively, the array is ordered, and particles are roughly uniform in size. In figure 3, the resulting polymer formation when a TL205 template was used can be seen. In contrast to when E7 was used, the polymer morphology contains a mixture of hexagonally ordered particles, stray particles, and large clusters of aggregated polymer. The particles were found to be notably smaller, with an average particle size of $\approx 1.3\text{ }\mu\text{m}$. All quantitative analysis of particles was performed using ImageJ/Fiji, and the “Analyze Particle” Function.

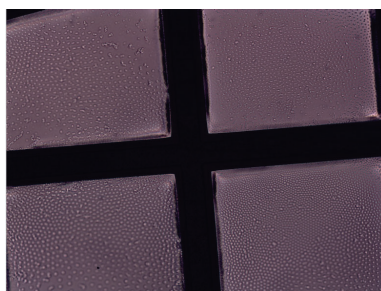


Figure 2: Resulting polymer formation when E7 was used as a template for polymer growth. Neat hexagonal array of polymer particles.

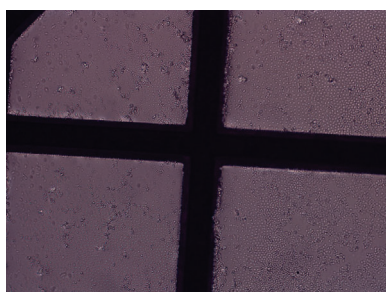


Figure 3: Resulting polymer formation when TL205 was used as a template for polymer growth. Less ordered array of polymer particles. Polymer clusters can also be seen.

Concentrations with my deposition conditions	E7	TL205
Concentration of DVB in LC	0.301 M	0.429 M

To confirm results, iCVD was repeated with varying parameters, such as filament temperature, initiator/monomer ratio, and deposition time. In every trial, the same pattern emerged: When TL205 is used as a template for polymer growth, the result is smaller polymer particles as well as a much less ordered arrangement of particles. Since this pattern

is consistent across many varying parameters, the cause for these differences was investigated. Given the fluorinated nature of TL205, it was hypothesized that the monomer, DVB, is less compatible with the TL205 than E7 and there is likely a lower concentration of DVB in TL205 than E7, which would result in smaller particles in TL205 as well as decreased order. This was tested through a two-part solubility test that relies in Henry’s Law, meaning that the pressure of monomer above the liquid crystal is directly proportional to the concentration of monomer inside the liquid crystal. The conclusion of this study revealed that contrary to the hypothesis, there is a higher concentration of DVB in TL205 than E7. Given the specific parameters of the depositions used in this study, the concentration of DVB in TL205 is 0.429 M, while the concentration is only 0.301 M in E7. While monomer solubility certainly affects polymer formations, more testing needs to be done to determine the cause for the decreased particle size and disorder that is seen when TL205 is used as a template for growth.

Conclusions and Future Steps:

When TL205 is used as a template for polymer growth, smaller polymer particles are formed, and formations are generally more clustered and chaotic than when E7 is used as a template for polymer growth. The reason for this is not due to increased monomer solubility in E7 compared to TL205 as we hypothesized, and more testing of LCs needs to be done to determine the reason for the differences in polymer morphology.

Figure 4: Results of solubility test.

References:

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