

Abstract

Polycaprolactone (PCL) is a biodegradable, semicrystalline polymer used in various biomedical applications, most notably in drug delivery. Upon crystallization of the polymer, PCL forms spherulitic crystal structures, with these structures sizes being confined under thin film casting. PCL films have previously been cast utilizing flow coating methods, with film thickness being controlled by casting velocity. PCL has previously demonstrated film dewetting after melting and recrystallizing, showing a potential for the extent of dewetting being dependent upon film thickness and PCL molecular weight. The dewet portions of thin films represent displaced PCL due to the difference in surface energies of the substrate and polymer material. Built upon previous studies of PCL dewetting, this project investigates a correlation between polymer dewetting and both thickness and molecular weight.

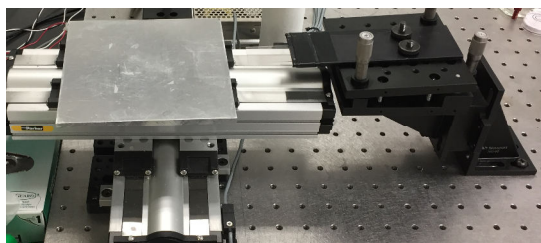
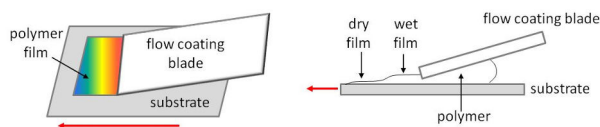
Materials/Methods

1) Solution preparation:

- Solutions were prepared in 20 mL vials in different weight solutions with regards to the amount of PCL compared to toluene. After initial stirring, the solutions were filtered into another 20 mL vial.

2) Flow coating:

- Cast various solutions onto silica substrate
- Glass slide drags solution across substrate of pre-determined length
- Stage is moved left while glass slide remains stationary



3) Optical microscope (below left):

- Bright field (below right) and dark field (below middle) images taken at 10x, 20x, 50x
- Images taken for both as cast and melted and recrystallized films

4) Melt and recrystallize:

- Melt at 100 °C for 10 minutes
- Recrystallized at 25 °C for 30 minutes
- Take optical images to determine dewetting

Results

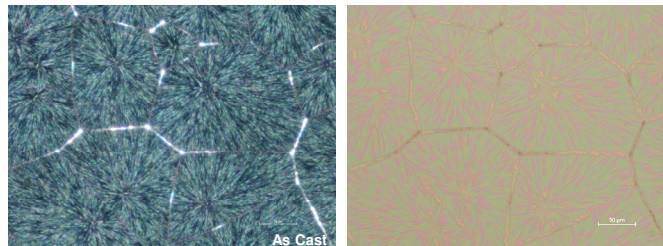


Figure 1: As cast images of film thickness ~850 nm at 20x in dark field (left) and bright field (right).

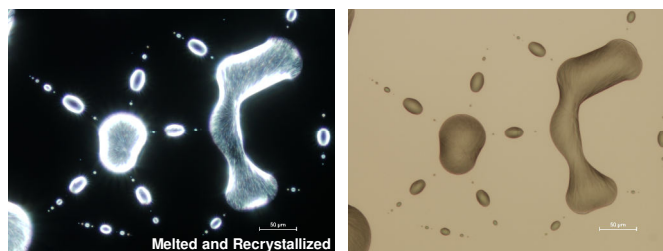


Figure 2: Melted and recrystallized images of film thickness ~850 nm at 20x of Figure 1 images in dark field (left) and bright field (right).

- No dewetting observed in as cast films
- Dewetting observed upon melting and recrystallization
- Bright field and dark field images used for analysis
 - Bright field helps confirm dewetting
 - Dark field used for quantifying the amount of dewetting

ImageJ Analysis of Dewet PCL

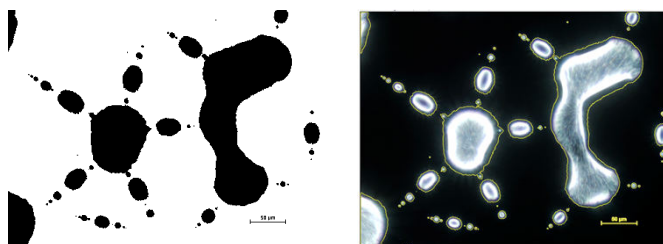


Figure 3: ImageJ analysis on dark field melted and recrystallized image from Figure 2 in black and white on the left and selected polymer for measurement on the right.

- Above images strictly used to develop analysis process
- ImageJ analysis selected area of non-dewet polymer after melting and recrystallization for 50x images
- Total Area - Selected Polymer Area from ImageJ = Dewet area
- Area measured in pixels and can be further converted to metric units

Discussion

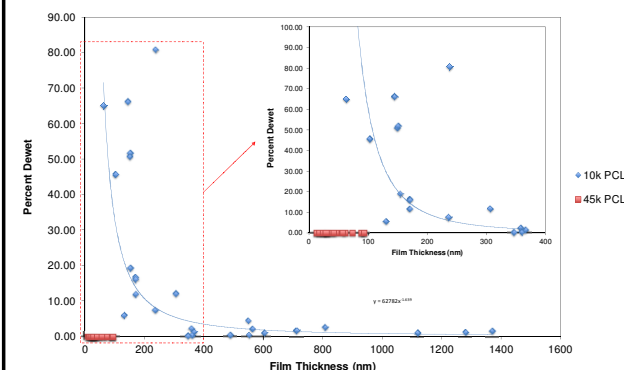


Figure 4: Charts representing the variation of film dewetting upon melting and recrystallizing with film thickness or 10k PCL. Inset chart shows smaller range of film thicknesses.

- Percent dewet v. thickness comparison
- Downward trend in dewetting as film thickness increases
- Levels off to no visible dewetting around 400-500 nm
- Large jumps in dewetting within hundred nanometer increments

Future Work

- Using AFM and profilometer to evaluate grain boundaries and measure extent of dewetting
- Investigating film acceleration with film dewetting
- Investigation of different PCL molecular weights

References

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