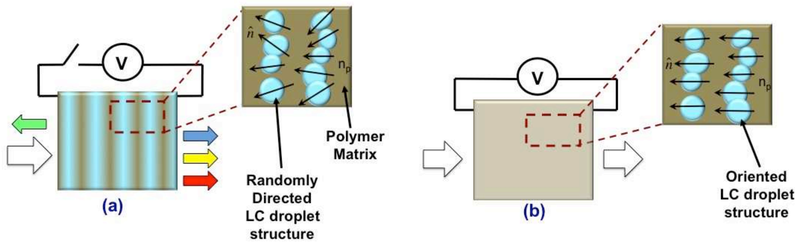
Variable Pressure Mode SEM Studies of Liquid Crystal Droplets

Kashma K. Rai and Adam K. Fontecchio

Department of Electrical and Computer Engineering, Drexel University, Philadelphia, PA, USA

Introduction



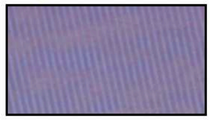
Reflection mode Holographic Polymer Dispersed Liquid Crystals. (a) No voltage applied state (b) Voltage Applied State

- Holographic Polymer Dispersed Liquid Crystals (HPDLCs) are electro optic thin film which consist of liquid crystal (LC) droplets dispersed in polymer matrix. They are arranged in stratified planes.
- HPDLCs are formed by optical interference of laser beams. Monomer units phase separate and polymerize in the bright regions of the interference pattern whereas the liquid crystals diffuse in to the dark regions forming droplets.
- Depending on formation setup reflection and transmission holograms are formed.
- Reflection mode HPDLCs reflect a particular wavelength depending on the grating spacing
  - Application: Electro optic devices such as optical switches, reconfigurable lenses
- Transmission mode HPDLCs are diffraction gratings. They diffract incident intensity depending on incident wavelengths
  - Application: Wavelength filtering devices such as dispersive spectrometer

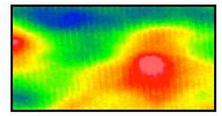
Morphology Study

In this work the liquid crystal(LC) droplet/polymer layers of HPDLCs are imaged using Variable Pressure SEM for the first time.

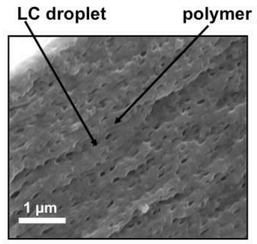
- An in-depth understanding of the morphology such as the grating structure is essential to understand optical characteristics and improve the diffraction efficiency of HPDLCs.
- Methods such as polarizing optical microscope and profilometer are convenient for the observation of the HPDLC grating without the need for elaborate sample preparation.
- Feature sizes in the micrometer range can be viewed with ease. A HPDLC film of ~3 µm grating pitch was easily viewed as shown using POM imaging and profilometer.



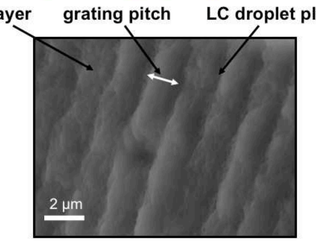
POM image of a HPDLC with ~3 µm grating pitch



Surface profile of HPDLC with grating pitch of ~ 3 µ m



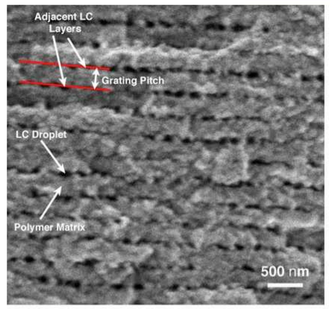
SEM image of reflection type HPDLC with a collapsed polymer matrix after the removal of LCs.



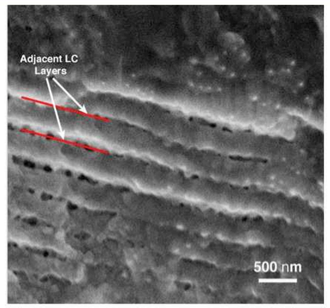
SEM image of the transmission type HPDLCs

- SEM is an incredibly useful tool to study the nanometer scale structure of HPDLCs. **SEM technique is indispensable for inspection of phase separation between LCs and polymer, the LC droplet diameter, droplet structure and thickness of the grating pitch.** HPDLCs are typically imaged using the high vacuum mode. Here we have used the variable pressure mode for imaging them for the first time.
- The advantages of the variable pressure SEM over high vacuum SEM are
- The polymer LC composites are non conductive. In the high vacuum mode they are coated with a conductive layer of platinum to prevent accumulation of charge on the surface. They do not need any pretreatment with a conductive layer in the variable pressure mode enabling faster preparation time of the SEM samples.
- The LC have to be removed prior to imaging using high vacuum SEM. This sometimes leads to the collapse and deformation of the grating structure. In the variable pressure mode the HPDLC samples can be imaged with the LC intact. This enables imaging the true grating morphology.

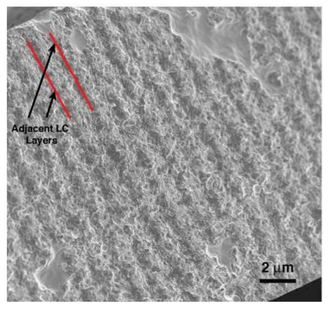
Comparison of Variable Pressure and Hi-Vac SEM Imaging



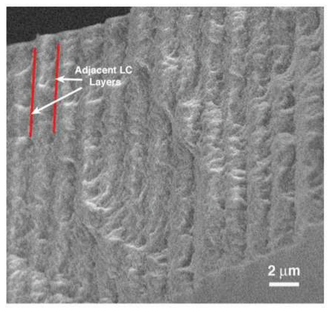
High Vacuum SEM image of reflection mode HPDLC



Variable Pressure SEM image of reflection mode HPDLC



High Vacuum SEM image of transmission mode HPDLC



Variable Pressure SEM image of transmission mode HPDLC

Grating Pitch= thickness of one layer of LC and polymer or the distance between the centers of adjacent LC layers as shown in the SEM images

Recipe

- Reflection and Transmission mode HPDLCs were fabricated using acrylate recipe consisting of
  - -45% blend of tri-functional and hexa-functional oligomers 4866 and 8301 respectively from Cytec
  - -32.4% Liquid Crystal BL038 from Merck
  - -12.4% initiation made of 4% Rose Bengal, 10% N Phenyl Glycine and 86% of N-Vinyl Pyrrolidone
  - -10% surfactant Tween 80 for improving the electro optic response of HPDLCs.
- Zeiss Supra 50VP SEM was used to image the HPDLCs.

SEM Image Analysis

- Analyzing the SEM images, a reduction in the grating pitch by 18.6% for the high vacuum image compared to variable pressure image of the reflection mode HPDLC was observed.
- Comparing the transmission mode SEM images a reduction in the grating pitch by 17.5% was observed.
- This is due to the removal of LC in high vacuum SEM imaging, which cause the polymers around the removed LC regions to contract, whereas the variable pressure SEM image reflects the true grating morphology due to non removal of LC.
- These SEM images of the HPDLC cross-section provide information on the LC size and shape distribution. As seen from the reflection HPDLC SEM images the LC droplet shape on an average is elongated along the layers and the LC nanodroplet size and shape is quite variable. The transmission HPDLC images clearly indicate the polymer having rough edges at LC/ polymer interface.

Conclusion

- HPDLCs were imaged for the first time using scanning electron microscope in the variable pressure mode, without LC removal and without conductive coating of Pt-Pd.
- A comparison of the grating spacing using image analysis tool between the two types of imaging showed an average decrease of grating pitch by 18.6% for reflection HPDLC imaged in high vacuum compared to variable pressure mode. A decrease of grating spacing is observed for transmission HPDLCs by 17.5% for these two respective modes. This suggest the contraction of the grating pitch due to the removal of the LC droplets
- The accurate grating morphology of the the polymer/LC composites using variable pressure SEM enables accurate electro-optical characterization. It also provides exact parameters for theoretical modeling of HPDLCs.

Acknowledgements

- NASA New Investigator Program Award No. NNG04G042G
- NSF ECCS 0621811
- We are grateful to the help extended by the Drexel Centralized Research Institute and extend our special gratitude to Edward Basgall and Dee Breger

Contact: kashma@drexel.edu, fontecchio@drexel.edu