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PI Name:	Yokoyama, Hiroshi Ph.D.		
Project Title:	Structure and Dynamics of Monodisperse Liquid Crystal Domains created on Suspended, Molecularly-Thin Smectic Films using Sub-Femtoliter Inkjet Technology		
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PI Email:	hyokoyam@kent.edu	Fax:	FY
PI Organization Type:	UNIVERSITY	Phone:	330-672-2633
Organization Name:	Kent State University		
PI Address 1:	Glenn H. Brown Liquid Crystal Institute		
PI Address 2:	1425 Lefton Esplanade		
PI Web Page:			
City:	Kent	State:	ОН
Zip Code:	44242-0001	Congressional District:	13
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Contact Monitor:	Tin, Padetha	Contact Phone:	216-433-8164
Contact Email:	Padetha.Tin@grc.nasa.gov		
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Key Personnel Changes/Previous PI:	November 2018 report: No change during this term.		
COI Name (Institution):	Emelyanenko, Alexander Ph.D. (Co-PI/ Lomonosov Moscow State University) Tabe, Yuka Ph.D. (Co-PI/ Waseda University, Japan)		
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Task Description:

Liquid crystals are the unique state of matter in which the molecular order existing in the solid state can persist even in the flowing liquid state. Understanding the molecular interactions underlying the microscopic order is crucial for further development of better performing liquid crystals for industrial applications and also for elucidating the molecular mechanisms of a wide range of biological structures. The goal of this project is to study the evolution of microscopic islands of liquid crystals configured on a thin liquid crystal film in the microgravity environment. The islands are deposited on the film by means of a novel sub-femtoliter inkjet device in a prescribed configuration. The time dependent changes of configuration and the rotational motion of islands will reveal the hidden molecular action responsible for the liquid crystallinity. The research effort in 2018 has been focused on further development of the sub-femtoliter inkjet technology, continuing the progress made in 2017. The deposition performance has been quantified and confirmed the droplet sizes below 10fl by optimizing the voltage waveform and the gap between the nozzle and the target. A continuous as well as drop-on-demand deposition of viscous liquid crystals has been realized both solid targets and free standing smectic films. Several different versions of device for automatic preparation of free standing smectic films have been developed and tested for use in the automated hardware in microgravity.

Rationale for HRP Directed Research:

Research Impact/Earth Benefits:

The principal scientific objective of this flight experiment is to explore the hidden phenomena in free standing smectic liquid crystal films in the microgravity environment in which the capillarity-induced forces disappear. Generally, interactions of microscopic particles and islands freely-suspended in two-dimensional liquid films are of central significance in a wide range of industrial fields ranging from oil and mineral recovery, food processing, pharmaceuticals, coating and wet processes, as well as in basic sciences dealing with protein-protein interactions in cell membranes. In Earth's gravitational field, intricate molecular interactions are often overwhelmed by capillarity forces and are hardly accessible in direct physical experimentations. Microgravity in space enables us to approach these phenomena such as Ostwald ripening, molecularly mediated island-island interactions and the Lehman rotation in liquid crystalline islands and films driven by the transmembrane molecular flow through the observation of configurational evolution of liquid crystalline islands on the smectic thin film. To prepare the required initial arrangement of islands, we develop and employ the sub-femtoliter inkjet deposition technology. Crucial for the liquid crystal science and technology is the understanding of intermolecular interactions responsible for the formation of liquid crystal phases. This space research aims to provide novel information that is hardly acquired from ground-based experiments.

Femtoliter Inkjet

We have continued the development of sub-femtolitter inkjet device for use with viscous liquid crystals with a view to establishing the level of stability, reliability, and controllability in terms of the size of droplet, the position and timing of deposition. The common technical challenge in the inkjet technology is to avoid the clogging of nozzles. Since clogged nozzles are fatal in the space experiment, our design employs an open cell made of a 1mm-diamater capillary tube with a fine tungsten electrode (~10 microm-diameter) at the center. The ink liquid wets the tungsten electrode, forming a meniscus. Application of high bias voltages (<1kV) induce the electrowetting behavior, which attracts more ink liquid to toward the apex of the electrode. Above a certain threshold voltage, which depends on the height and the shape of the tungsten electrode, there occurs a sufficient supply of ink liquid to the tip of the electrode, starting the electrostatic deposition of the ink. Using a pulsed voltage, we confirmed that a fine droplet can be deposited at a rate up to 100 droplets/sec. Under application of a relatively low DC voltage compared to the pulsed operation, a continuous deposition with a controllable rate is also possible.

The experimental setup for quantitative real-time observation of inkjet deposition of islands on free standing smectic films consists of a pair of stereo microscopes, installed in such a way that the free standing film and the inkject nozzle can be observed simultaneously from both top and side directions. The top view microscope is equipped with a top down illumination to image the smectic film in the reflected light microscopy (RLM) mode. The RLM allows determination of the film thickness from the color and contrast of the image.

We observed the deposited droplets of ink cumulatively obtained by 100 times pulsed-deposition of liquid crystal 8CB on an ITO glass substrate. From the total volume of the liquid crystal deposited, the volume of individual inkjet droplets is estimated to be at most 20 femtoliter. We observed a clear tendency that the lower the bias voltage, the smaller the minimum droplet size becomes, reaching even sub-femtoliter. However, the deposition condition becomes progressively unstable as the bias voltage is decreased. Moreover, at high pulse voltages over 1.5 kV, deposition of multiple drops often happens. We suspect that at such high voltages, the droplet is so much charged that the surface tension of the ink can no longer be large enough to keep the integrity of the droplet. The over charged droplet splits into pieces creating the multiple droplet deposition.

We successfully carried out the deposition of liquid crystal islands deposited on a free standing semectic film using the electrostatic inkjet. The initial size of the island is 20 microm~30 microm in diameter, and the islands rapidly coalesce with the neighboring islands, forming larger domains. Since the droplet before reaching the film is too small to observe even at the highest magnification, the process of island deposition appears as if the island is suddenly born and grows until it reaches the equilibrium thickness and size. An apparent drawback of the electrostatic deposition is that the electric force due to the voltage pulse vibrates the film and disturbs the configuration of the deposited islands. In order to prevent this behavior, it is necessary to install an extraction electrode held at the same potential as the free standing

In order to assess the effect of extraction electrode upon deposition of liquid crystal islands on a free standing smectic film, we have set up a simple extraction electrode. The electrode has a 200 microm-wide gap, through which the deposition can be made, and is placed about 0.5 mm from the film. We could confirm that the electrostatic vibration of the film was completely suppressed, and a fairly gentle deposition of islands was made possible. However, the operation of the extraction electrode is not as yet as reliable as it should due to the required delicate control of the trajectory of the ejected droplet. In the open cell design, the meniscus around the central electrode is not perfectly stable, thereby causing unpredictable fluctuation of the deposition conditions. To eliminate the influence of meniscus, we are currently developing a new design that is to electrically shield the meniscus.

Design and Implementation of Film Drawers

Automated preparation of free standing smectic films is a key hardware requirement for our experiments in space. The conventional method is to drill a hole in a glass slide and manually spread the film over the hole with a razor blade from

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a small amount of liquid crystal applied to the edge of the hole. Although this method works well in the ordinary lab environment, it is difficult to establish a level of repeatability and accuracy of the film thickness as required in the present space experiment. Furthermore, it is not straightforward to prepare a free standing film in such a way as to separate two isolated compartments for Lehman rotation experiments.

The earliest versions of the film drawer were designed such that instead of the razor blade, a 20 mirometer or so thick plastic or metallic sheet with a large hole is traversed between the metal holders across the hole. With the liquid crystal applied at the edge of the hole, the free standing film grows smoothly as the exposed area through the sliding sheet expands. The operation is quite stable and reproducible, yet the drawback is the difficulty to avoid the self-thinning of the free standing film as the liquid crystal is sucked into the narrow gap between the metal plate and the sliding sheet. After one day or two, the film becomes almost two molecular layers thin everywhere, making it rather fragile. A more recent version of the film drawer with the improvement in the thinning action utilizes a thin tungsten wire instead of the sliding sheet to draw the film from the knife edge. Since the contact area between the wire and the cover plates is much smaller than that in the sliding sheet-based drawer, the free standing film is much less likely to sucked back into the gap as before. The drawback of this design, however, is the relative difficulty in driving the wire. Further improvement is currently underway.

Additional Activities

On April 24 and 25, 2018, the Science Concept Review was held at NASA Glenn Research Center inviting external reviewers. From our team, Yokoyama and Tabe attended the meeting and discussed the basic idea, the current status, and the remaining challenges of the project. Due to a visa problem, Emelyanenko could not attend the meeting in person, but participated in discussions using an online conference. The review committee was of the opinion that the scientific goal and approach of the project is scientifically quite meaningful, yet the hardware, especially the femtoliter inkjet must be more sophisticated to realize the true on-demand deposition of femtoliter droplets without disturbing the free standing film.

Graduate Students: Following graduate students at the LCI & Chemical Physics Interdisciplinary Program contributed to the work described: Mengfei Wang, Joseph Angelo, and Wei Chen.

Future Plan: The ground based scientific activity will continue to establish the sub-femtoliter inkjet technology up to the level that electrically neutral droplets of variable sizes from submicron to tens of microns can be deposited with negligible disruption on the part of the smectic films. We have made a concept design of the ultimate setup with symmetrically positioned ejection and guard electrodes. Precise microfabrication of the extraction gate electrode will be the next step along with the development of neutralizing mechanism. Precise quantification of the droplet volume and charges must be made in order to identify the optimal deposition condition. In addition, it is necessary to further develop a reliable technique to reproducibly prepare a free standing smectic film and control the film thickness by external voltages to allow Lehmann rotation studies. A compact reflected light microscopy will be developed together with a real time image analysis system to monitor the two dimension distribution of the film thickness.

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