

<b>Fiscal Year:</b>	FY 2020	<b>Task Last Updated:</b>	FY 12/09/2019
<b>PI Name:</b>	Yokoyama, Hiroshi Ph.D.		
<b>Project Title:</b>	Structure and Dynamics of Monodisperse Liquid Crystal Domains created on Suspended, Molecularly-Thin Smectic Films using Sub-Femtoliter Inkjet Technology		
<b>Division Name:</b>	Physical Sciences		
<b>Program/Discipline:</b>			
<b>Program/Discipline--Element/Subdiscipline:</b>	COMPLEX FLUIDS/SOFT MATTER--Complex Fluids		
<b>Joint Agency Name:</b>		<b>TechPort:</b>	No
<b>Human Research Program Elements:</b>	None		
<b>Human Research Program Risks:</b>	None		
<b>Space Biology Element:</b>	None		
<b>Space Biology Cross-Element Discipline:</b>	None		
<b>Space Biology Special Category:</b>	None		
<b>PI Email:</b>	<a href="mailto:hyokoyam@kent.edu">hyokoyam@kent.edu</a>	<b>Fax:</b>	FY
<b>PI Organization Type:</b>	UNIVERSITY	<b>Phone:</b>	330-672-2633
<b>Organization Name:</b>	Kent State University		
<b>PI Address 1:</b>	Glenn H. Brown Liquid Crystal Institute		
<b>PI Address 2:</b>	1425 Lefton Esplanade		
<b>PI Web Page:</b>			
<b>City:</b>	Kent	<b>State:</b>	OH
<b>Zip Code:</b>	44242-0001	<b>Congressional District:</b>	13
<b>Comments:</b>			
<b>Project Type:</b>	FLIGHT	<b>Solicitation / Funding Source:</b>	2015 NNH15ZTT002N MaterialsLab Open Science Campaigns for Experiments on the International Space Station
<b>Start Date:</b>	12/30/2016	<b>End Date:</b>	12/29/2021
<b>No. of Post Docs:</b>	0	<b>No. of PhD Degrees:</b>	0
<b>No. of PhD Candidates:</b>	1	<b>No. of Master' Degrees:</b>	0
<b>No. of Master's Candidates:</b>	0	<b>No. of Bachelor's Degrees:</b>	0
<b>No. of Bachelor's Candidates:</b>	0	<b>Monitoring Center:</b>	NASA GRC
<b>Contact Monitor:</b>	Hatch, Tyler	<b>Contact Phone:</b>	216.433.5073
<b>Contact Email:</b>	<a href="mailto:tyler.r.hatch@nasa.gov">tyler.r.hatch@nasa.gov</a>		
<b>Flight Program:</b>	ISS		
<b>Flight Assignment:</b>	Liquid Crystal Facility		
<b>Key Personnel Changes/Previous PI:</b>	November 2019 report: No change during this term.		
<b>COI Name (Institution):</b>	Emelyanenko, Alexander Ph.D. ( Co-PI/ Lomonosov Moscow State University, Russia ) Tabe, Yuka Ph.D. ( Co-PI/ Waseda University, Japan )		
<b>Grant/Contract No.:</b>	NNX17AD68G		
<b>Performance Goal No.:</b>			
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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 2019 has been focused on the two fronts: (1) Development to an automated film preparation system and an analytical tool for rapid mapping of the thickness distribution of the film, and (2) Improvement of the ultrafine inkjet device to allow better controlled deposition of droplets. The thickness of free standing films are quantized by the number of molecular layers and play a significant role in determining the structure and interaction of droplets and their structural evolution; For the Lehmann rotation, the sense and speed of rotation are known to undergo drastic change, and hence it is vital to characterize the film thickness in the first place. We have developed a novel approach for thickness measurement based on the color of the reflected light, not on the intensity of reflection, which has been used for decades as the standard technique. For 2D analysis of the film images, the color is much more reliable variable than the intensity. We also developed an automated film preparation hardware that can be considered a prototype for the ultimate hardware for flight experiments. In 2017 and 2018, we demonstrated the feasibility of ultrafine inkjet technology based on electrostatic deposition. We refined the device by employing a reproducible electrochemical preparation method of sharp metallic needles. The hardware is equipped with fine positioning mechanisms to adjust the relative positions of the needle, the extraction electrode and the smectic film target.

**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 to name a few. 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, which is integrated to an automated film preparation and observation hardware. 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.

**Task Progress:**

In the projected flight experiments, free standing films of typical smectic liquid crystals are prepared either in the flat film form or as an inflated bubble. The thickness of the film is self-regulated through the chemical potential of the liquid crystal molecules, which is controlled by electric field and/or pressure applied to the liquid crystal. On the smectic film will be deposited islands of distinct liquid crystalline material in a prescribed configuration by means of the electrostatic sub-femtoliter inkjet deposition device. Optical microscopy observation of the translational and rotational movement islands and the variation of island sizes is conducted and the results are compared with theoretical predictions based on a particular type of intermolecular interactions.

Based on the feasibility study conducted in the past two years, we have developed a prototype hardware integrating the ultrafine inkjet device and a motorized film drawer. The core of the hardware is enclosed inside a metallic chamber to allow application of moderate level of differential gas pressure outside and inside the film compartment. For the purpose of Lehmann rotation experiments, the film must be subjected to a gradient of partial pressure for certain molecules to be transferred; for this reason, the core has two separate closed compartments on both sides of the film. The newly developed film drawer consists of two metallic plates through which 10 mm-diameter hole has been drilled. The holes are tapered toward the outside so that the inner surfaces in contact to each other have a sharp knife edge. The inner surfaces are polished to allow a good surface contact of the plates. The smectic liquid crystal is applied to the inner surfaces, and the two plates are relatively slid in such a way that the two holes begin to open where the smectic film is suspended. The performance of the film drawer is highly stable and reproducible; Once a small amount of smectic liquid crystal is applied continuously to the contact area, the success rate for preparation free standing film is practically 100% except in such a case an excessive vibration is induced during spreading.

The lower compartment of the core hardware houses the ultrafine inkjet device. In the previous term of the project, we have used a cutoff edge of 10  $\mu\text{m}$  - 50  $\mu\text{m}$  thick tungsten wire as the inkjet tip. Due to the flexibility of wire, it was not possible to precisely position the Wire. To alleviate this difficulty, we employed in the new design an electrochemically etched tungsten wire. Starting with a 500  $\mu\text{m}$ -thick tungsten wire, the edge of the wire can be etched in KOH solution in 1 hr under the application of 3V DC voltage with the tungsten wire being the anode. To obtain the sharp edge, it is necessary to slowly extract the wire through the surface of the KOH solution. The rate of extraction determines the taper angle. The radius of curvature at the apex is well below 1  $\mu\text{m}$ . The sharp needle is then inserted in a glass capillary. This procedure is straightforward compared to the previous preparations for the rigidity of the main wire. The bottom end of the glass capillary is partly glued to the tungsten wire. The ink materials can be easily applied by capillary action. The inkjet nozzle thus prepared is then set inside the core hardware, which has 6 degrees of freedom x,y,z positioning system.

The thickness of the smectic film is a crucial factor in determining the behavior of smectic films. Hence, measuring the thickness distribution over the film is the basis for quantitative analysis and interpretation of experimentally observed results. A standard technique is to measure the reflectivity of monochromatic light from the film. This method works for a point wise observation, but accurate mapping of the film thickness is difficult for poor areal definition of illumination and the inevitable distortion of the film caused by an accidental trapping of the menisci at the rim.

Since the brilliant coloration is one of the unique features of the free standing smectic film as well known for soap films, we decided to make use of this color information rather than the reflected light intensity. Under white light illumination,

indeed, the molecular layer steps are clearly visible, and the discrete change of color results from the layer-by-layer thickness variation, indicating the possibility to estimate the thickness from quantitative analysis of color. Instead of a monochromatic light as in the intensity method, a stable broadband light source needs to be used for illumination of the film. For this purpose we use a 3-LED data projector as the light source for its outstanding stability and capability to computationally control the color and the intensity. There are three spectral peaks corresponding to the center color of the three LEDs, namely Red, Green, and Blue. This light from the projector is launched on the optical fiber bundle and is conveyed to one of the optical paths of a stereo microscope which has a zoom mechanism automatically optimizing the illumination beam size.

Based on the characteristics of the light source and the color sensitivity of the CMOS (complementary metal-oxide semiconductor) image sensor, we developed a theoretical color model as a function of the film thickness. The color information is extracted from the CMOS camera and is numerically compared with the theoretical model, yielding the thickness estimates. The relationship between color coordinates and the thickness is unique except for a few points and are confirmed to be used for precise determination of the thickness to the accuracy of molecular layer thickness around 3 nm or even better.

Bibliography Type:	Description: (Last Updated: 12/04/2023)
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