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IIIII The Kikuchi-Kossel Experiment - Colloidal Crystals under Microgravity IIIII
(Original Paper)

Development of a Colloid-Crystallization Observation Facility on the ISS/KIBO

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Abstract

We report on the onboard apparatus for the Japanese colloid "Kikuchi-Kossel" experiment on the ISS/KIBO. The system was devoted to the KIBO utilization research theme "Study on the mutual interaction process between colloids", as proposed by Prof. Sogami and started in 2011, where the main objective is observation of structure formation process in colloid dispersions under micro-G condition to verify existence of a long-range attraction force among the colloid particles. Based on the mission requirement, we have implemented a Kikuchi-Kossel laser-diffractometer and void-observation systems, where the former allows structural analysis of the colloid crystal structure and the latters allow the structure formation study at a mesoscopic (~10-100 μm) and macroscopic scale(~1mm) in colloid dispersions. The system also includes space-unique subsystems such as a stirring system and cuvettes etc. The paper summarizes system descriptions based on the Preliminary Design Review (PDR) phase. In December 2014, several colloid themes by other international partners are also planned for the ISS. It is expected that CCOF will contribute significantly to these colloid research community.

Keyword(s): CCOF, Kikuchi-Kossel, Void, Colloid, KIBO, International Space Station, JEM

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1. Introduction

The Japanese colloid research project "Kikuchi-Kossel", as proposed by Prof. Sogami (Kyoto-Sangyo Univ.) and started in 2011, is intended to extract universal information on a colloid-crystallization process by utilizing the ideal microgravity condition of 10^{-6} G on the ISS/KIBO, which is not attainable on the ground.

The main objectives include: (1) Study of crystal structures under micro-G conditions to verify the existence of a long-range attraction force to modify the standard colloid-crystallization theory "DLVO (Derjaguin - Landau - Verwey - Overbeek theory)", (2) Colloid informatics on the crystallization process using various colloid samples with their application to photonic crystal study [1)].

JAXA has been developing an onboard apparatus "Colloid-crystallization Observation Facility" (hereinafter referred to as CCOF) for this project, designed to be facilitated at Multi-purpose Small Payload Rack (MSPR) / Work Volume (WV) in KIBO. We have recently finished its Preliminary Design Review (PDR) [9)], whereby overall orientation of the system design was approved to meet the Interface Control Document (ICD) for the MSPR/WV; achieving a single-box component optimized to the MSPR/WV envelope as well as an operation design complying with the minimum usage policy on crew resources.

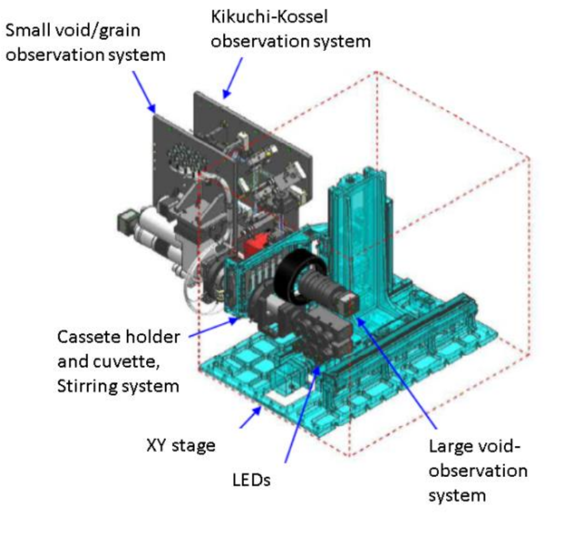

Fig. 1 Computer-Aided-Design for the CCOF. CCOF features a Kossel observation system, a small void/grain observation system and a large void-observation system.

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All operations except installation and sample changes can be conducted by ground operators, while ground-based researchers can acquire space experiment data through telemetry-commanding operations.

2. System descriptions

CCOF comprises the following subsystems:

(1) Kikuchi-Kossel observation system
(2) Small void/grain observation system
(3) Large void-observation system
(4) Cuvette
(5) XY-stage and stirring systems

The main components of the CCOF are shown in **Fig. 1**, while its specification is summarized in **Table 1**. These subsystems are described in subsequent sections.

2.1 Kikuchi-Kossel observation system

Kossel diffraction is known as a Bragg diffraction in a visible wavelength, as generated by mutual interaction between the incident laser and lattice constant of the colloidal crystal structure. These patterns appear as conics curves on the 2-dimensional plane and crystal structures can be retrieved from the pattern information and vice versa. [2), 3)] In our space project, the Kossel system is applied to studies on colloid-crystal structures [1), 5)].

Figure 2 shows a conceptual diagram for illustrating the optics of the CCOF/Kikuchi-Kossel observation systems.

The system is designed to detect the backward scattering of Kossel patterns. Within the system, a 1 mm^2 rod mirror having a tilt angle of 45 degrees on the area sensor plays key roles such as (1) in redirecting the incident laser 90 degrees, in the direction of the colloid sample at the cuvette, (2) in preventing from a CCD saturation by the counter reflection of the laser at the cuvette by concealing it, and (3) in earning the solid angle of the reflected Kossel patterns; enabling virtually complete detection of backward scattering.

Wavelengths of 405- and 640 nm semiconductor lasers are adapted for the incident lasers and a 24 mm ×36 mm CCD area sensor is adapted for the detector. The area sensor adapted in our project is both wide and has a high Signal-to-Noise Ratio (SNR) as well as a wide dynamic range to efficiently detect Kossel patterns, the brilliance of which varies in order from the scattering center to a position 60 degrees from the center.

Figure 3 shows an example of Kossel patterns using the engineering model. In our study, it emerged that Kossel patterns can be detected with appropriate brilliance of approximately 0.1 second, allowing the crystal structure and its trends to be retrieved at various points using the XY-stage. Note that the image shown here is processed by a software transaction featuring High Dynamic-Range Images (HDRI) [10)] of five different exposures, where these pictures are taken within the exposure time of 10 msec.

Table 1 Specification summary for the CCOF

Subsystem	Specifications
1. Kikuchi-Kossel diffractometer	・2 lasers (640- and 405 nm)for the light source. ・CCD area sensor (1024×800 pixels.) for the detection system ・Laser spot size on the cuvette : Product specification < 140 μm Achievement level (405 nm) 63 μm Achievement level (640 nm) 79 μm
2. Small void observation system	・Detect transmitting/scattering light from small void, grains (10-100 μm) ・End-to-End resolution: ・CCD sensor (130 mega pixels) ・Microscope: (2.5-30 times) ・Illuminators: (1) Coaxial illuminators for transmitting light through the void. (2) Ring-aligned illuminators deployed around the camera for grains, which can shed light in 3 different angles with five different colors. (400-700 nms)
3. Large void observation system	・Detect fluctuations such as clusters, crystal grains at a macroscopic scale (~1mm) ・CCD sensor (490 mega pixels) ・Resolution: 40 μm on the object. ・Also functions as a monitoring camera
4. XY-stage	・Used to control the target sample positions ・Stage Accuracy: Product specification 100 μm Achievement level 5 μm
5.Stirring system	・To stir the solution of colloids by a stirrer bar in a contactless manner
6.Cuvette and Samples	・50 colloid samples (Aqueous dispersions of polystyrene, those of silica, those of titania particles) ・Quartz glass cuvette 56(L)×12.5(W)×10.8(φ), (D):1(0.3) mm
7. Dimensions	941×320×380 mm
8. Weight	45.0 kg
9. Wattage	250 W at max.
10. Crew time	Estimated less than 8 hrs. (at PDR)

In the project, various colloid samples such as aqueous dispersions of polystyrene, those of silica, and those of titania particles are used to establish colloid informatics as well as its application to the study of photonic crystals.

2.2 Small void/grain observation system

The small void/grain observation system focuses on the ordering formation process in colloidal dispersion on a mesoscale of 10-100 μm. [4), 6), 7)] The small void/grain observation system comprises a CCD camera with a microscope and illuminators.

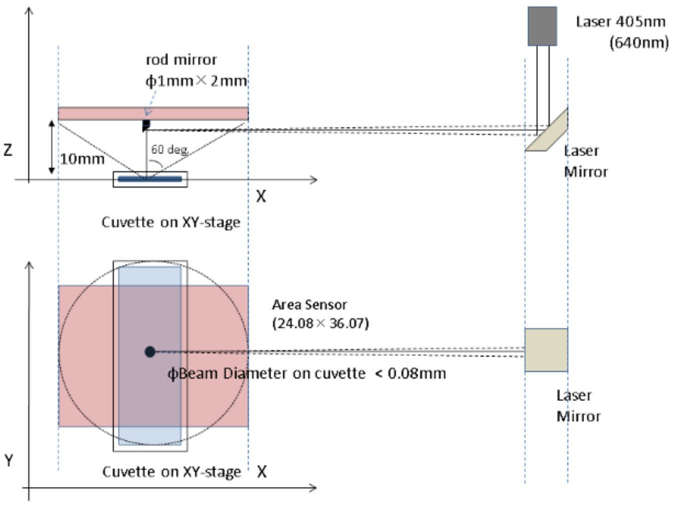

Fig. 2 Conceptual diagram for the optics of the CCOF/Kikuchi-Kossel observation system.

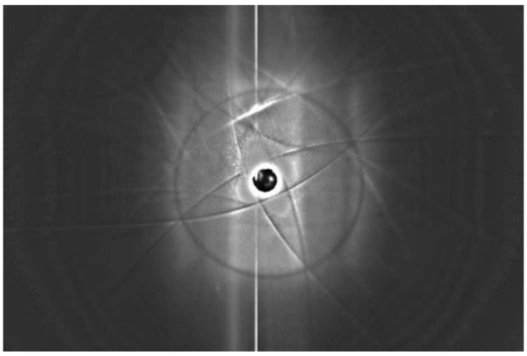

Fig. 3 Kossel image taken by the engineering model (Laser 405nm) Kossel diffraction patterns are visible at the center of the image, while a black circles at the central part corresponds to the shadow of the rod mirror.

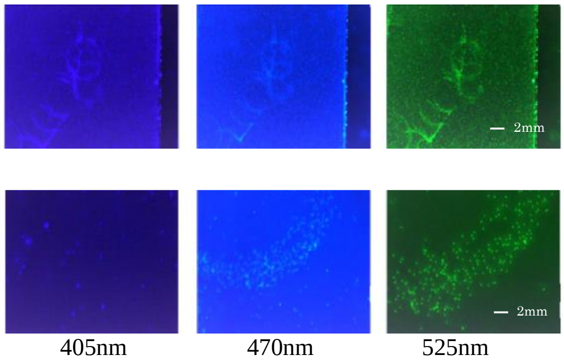

Fig. 4 Observed images taken by the small void/grain observation system. Images are taken with a 2.5 times microscope and with ring-illuminators of 405, 470 and 525 nm, respectively.

With reference to the CCD camera, a 130-megapixels CCD is used for the sensor with a 30 times or a 2.5 times microscope, which corresponds to a field-of-view of 0.2-2 mm and spatial resolution of 10-100 μm. The focal position of the camera can be adjusted with steps of 0.1 mm and a depth-of-field 1.1 mm relative to the object, allowing the void structure in the cuvette to be estimated by the focusing process. The camera can be remotely controlled and observed images can be downlinked at a speed of one image/sec.

The system has two types of illuminators, (1) ring-illuminators to uniformly illuminate the object and (2) coaxial illuminators to detect changes in density due to varying permeability. These illuminators comprise multiple LED colors between 400 and 700 nm, allowing grains to be detected based on the spectroscopic response to each color. The ring-illuminator deploys 10 LEDs around the optical axis, which will then be used to detect grains efficiently by utilizing the scattering property depending on the grain direction.

Figure 4 shows examples of images acquired by the small observation system, magnified 2.5 times by a microscope and with ring-illuminators at 405, 470 and 525 nm, respectively. It emerged that the small grain observation system has sufficient spatial resolution to detect the nature of the Brownian motion of the colloid sample, which is also considered sufficient to study the mesoscale crystallization process. [7]

2.3 Large void-observation system

The large void-observation system focuses on observing fluctuations such as clusters and crystal grains on a macroscopic scale (～1 mm.)[4), 8)]. With reference to the system, a 490 mega-pixel CCD is implemented as the camera sensor, with a viewing angle of 15 degrees, corresponding to spatial resolution of 40 μm on the cuvette, and with depth-of-field of 1.1 mm to be used to separate large void structures in the cuvette. A

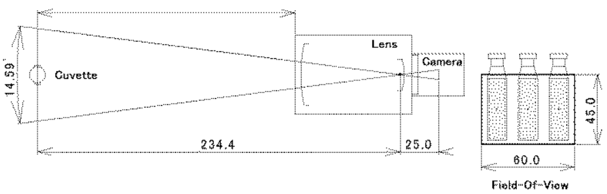

Fig. 5 Large void-observation system and its field-of-view on the stage. The large void-observation system has a 15-degree viewing angle, which allows simultaneous observation of three cuvettes. Using a black background board allows the object to be observed in high contrast with the ring-illuminator.

ring-illuminator is implemented to visualize object-appropriate contrast using a background board in black.

2.4 Cuvette

The colloid samples used in this project are enclosed in the cuvette and two types of cuvette (0.3 mm- and 1.0 mm depth) are designed selectively for the experiment objectives as shown in **Fig. 6**.

The 0.3 mm-type is used for transmission observations, while conversely, the 1.0 mm-type is used for macroscopic and Kossel observations. They are made of quartz glass, enclosing a stirrer bar covered in Teflon, which moves with the magnet in contactless manner. The cuvette is sealed with a Teflon seat and various fillers to retain sealability, and fixed by the aluminum cap by the climper. With reference to sealability, the sample weight can be retained to within approximately 99.98 % of its original weight or more for three months.

Five cuvettes are accommodated in a cassette holder to minimize the crew resources required on installation. During transportation and stowage, the cassette holder is covered airtight with another sealer to prevent any cracking of the glass and subsequent leakage of the colloid sample as well as filled with nitrogen gas, which remains inert relative to the sample and avoids any decline in the same. Due to these properties, the cassette holder can also be designed to use in highly toxic samples used in future missions.

2.5 XY-stage and Stirring system

CCOF has a relatively accurate XY-stage in positioning samples, which plays a key role to observe time variation of the colloid crystal during operation. Based on a preset, the experimental sequence with an accuracy of 5 μm, is used for re-positioning. Note that the XY-stage also helps downsize the system because it allows a flexible configuration at a 3-dimensional level.

A stirring system is implemented in the CCOF as discussed.

The spontaneous crystallization process means the crystals must be collapsed for an experimental reset of the space experiment. An iron bar covered in Teflon inside the cuvette

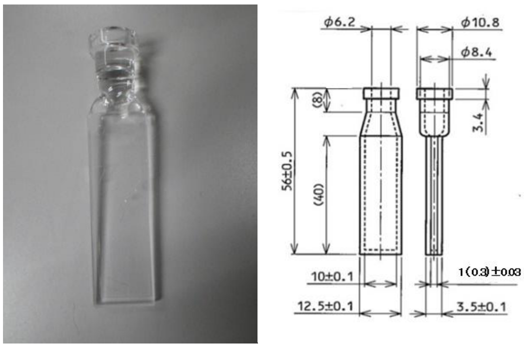

Fig. 6 The cuvettes used in this project (0.3 / 1.0 mm depth). They are made of quartz glass and enclose a stirrer bar.

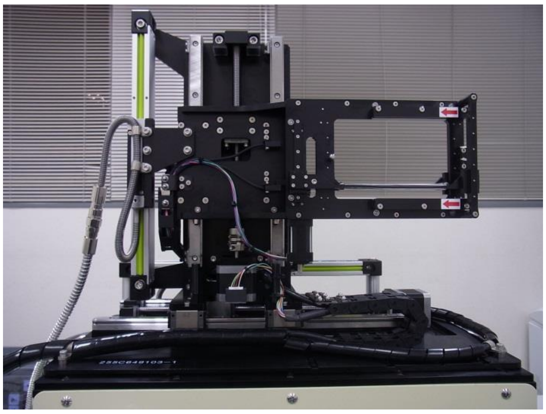

Fig. 7 The XY-stage and the stirring system XY-stage have accuracy of 5 μm for re-positioning. The stirring system stirs to initialize the colloidal dispersions by moving the stirrer bar in alignment with the up-down motions of the magnet.

functions as a stirrer bar and is considered neutral relative to the colloid sample due to the Teflon. It can be controlled by the magnet in a contactless manner, whereby the colloid sample can be initialized by the up-down movement of the stirring system. The cassette holder and attaching stirring system is shown in **Fig. 7**.

3. Summary

This paper summarizes the specification of the onboard apparatus for the Kikuchi-Kossel, "CCOF". CCOF features the Kikuchi-Kossel diffractometer and the void-observation systems, such as microscopes on a mesoscale and a macroscopic scale. And a compact XY-stage and a cuvette cassettes are specifically designed for the space experiment.

The system with its Kikuchi-Kossel diffractometer is appealing in terms of its uniqueness in analyzing the structure of colloid crystal. Further appealing features of the project include the use of various colloid samples such as aqueous dispersions of polystyrene, those of silica, and those of titania particles to establish colloid informatics as well as its applicability for studying photonic crystals. Note that it will also be easy to use CCOF for other colloid experiments in future, due to the simplified I/F and the cuvette design.

CCOF will contribute significantly to the colloid research community and function as a valuable platform for colloidal study, even for future missions.

Acknowledgments

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References

1) I.S. Sogami: Int. J. Microgravity Sci. Appl., **32** (2015) 320201. (in Japanese)
2) I.S. Sogami, T. Shinohara, M. Tanigawa, K. Ito and J. Yamanaka: Int. J. Microgravity Sci. Appl., **32** (2015) 320202. (in Japanese)
3) M. Tanigawa, T. Shinohara, J. Yamanaka, K. Ito and I.S. Sogami: Int. J. Microgravity Sci. Appl., **32** (2015) 320204. (in Japanese)
4) K. Ito, M. Ishikawa and I.S. Sogami: Int. J. Microgravity Sci. Appl., **32** (2015) 320206. (in Japanese)
5) C. Kakihara, J. Yamanaka, T. Shinohara, M. Tanigawa and I.S. Sogami: Int. J. Microgravity Sci. Appl., **32** (2015) 320205.
6) Y. Kataoka: Int. J. Microgravity Sci. Appl., **32** (2015) 320208. (in Japanese)
7) B.V.R.Tata: Int. J. Microgravity Sci. Appl., **32** (2015) 320209.
8) M. Ishikawa, S. Ito, R. Kitano, S. Miyoshi, and J. Habasaki: Int. J. Microgravity Sci. Appl., **32** (2015) 320207.
9) K. Kogure, T. Sone, and Y. Ito: Int. J. Microgravity Sci. Appl., **32** (2015) 320210. (in Japanese)
10) E. Reinhard, W. Heidrich, P. Debevec, S. Pattanaik, G. Ward and K. Myszkowski: High dynamic range imaging, acquisition, display, and image-based lighting. Morgan Kaufmann (2010).