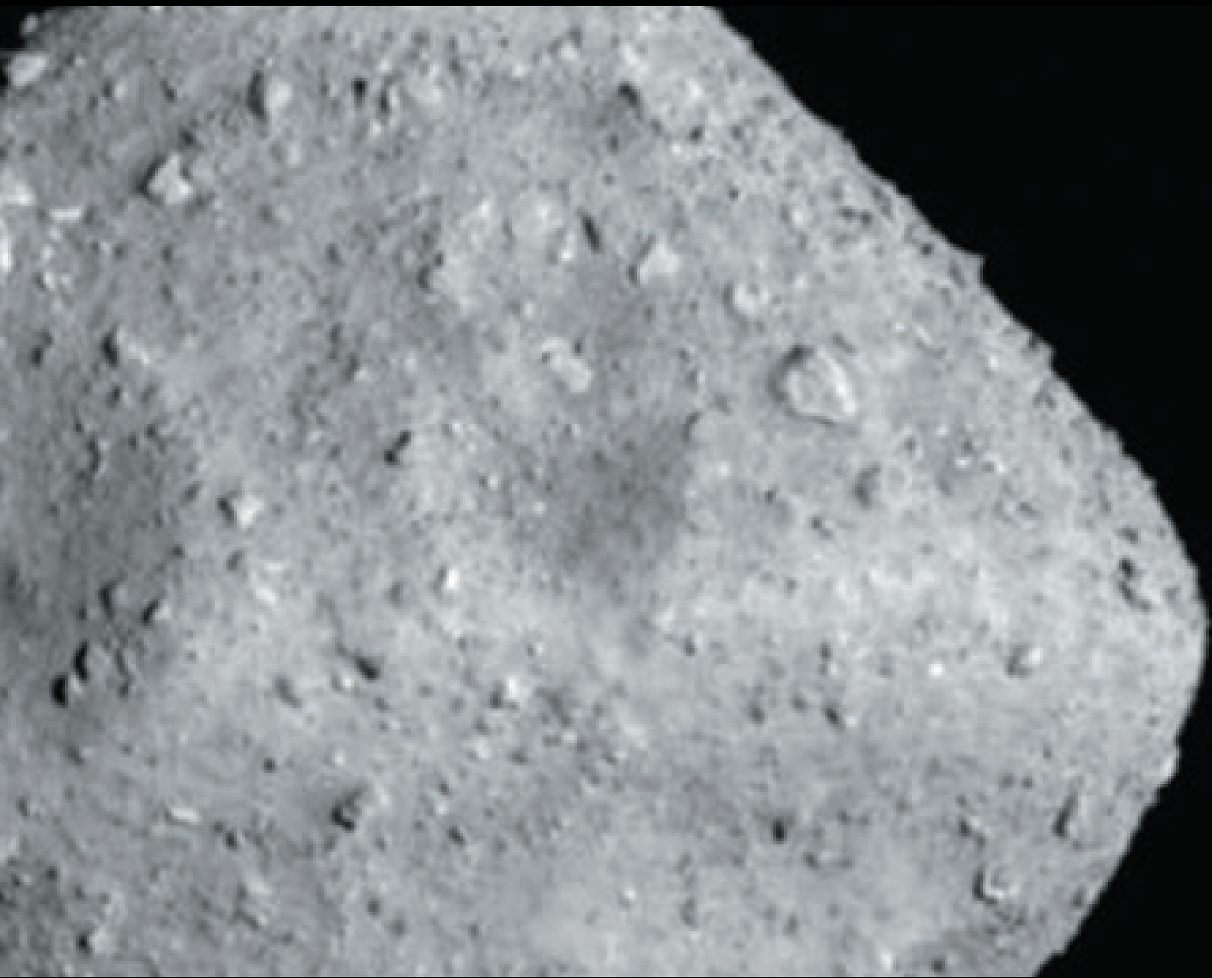
Synchrotron Radiation Analysis Reveals Material Structure of Asteroid Ryugu

Camera application case study



HAMAMATSU
PHOTON IS OUR BUSINESS

A clue to solving the mysteries of the origin and evolution of our solar system and the birth of life

Asteroid Ryugu, is thought to still contain water and organic compounds from 4.6 billion years ago when our solar system likely formed. The Japanese spacecraft Hayabusa2, launched in December 2014, retrieved grains from the asteroid as samples for analysis. The collected samples arrived in a special capsule on Earth in December 2020 and were handed over to professional researchers. Since then, continued analysis has revealed various facts that could not have been discovered from observatories on Earth.

We interviewed Dr. Uesugi of the Japan Synchrotron Radiation Research Institute (JASRI), who was in charge of analyzing the samples, about the methods and results of the analysis, as well as future prospects.

The interview was conducted in March 2023.

Dr. Kentaro Uesugi

Chief Scientist, Coordinator
Scattering and Imaging Division
Center for Synchrotron Radiation Research
Japan Synchrotron Radiation Research Institute (JASRI)

Born in Saitama, Japan, in 1973, Dr.Uesugi graduated from the School of Science at the Tokyo Institute of Technology and entered graduate school at the same university. During his PhD studies, Osaka University offered him a research position at JASRI, which operates and maintains SPring-8, a large synchrotron radiation facility. Since then, Dr.Uesugi has been continuously involved in everything from designing and fabricating experimental apparatus to conducting experiments, writing papers, and giving presentations to meet the global demand to “look inside with X-rays.” More recently, his analysis of the Ryugu grains brought back to Earth by the Hayabusa2 asteroid probe has attracted significant attention. In addition to being a chief scientist at SPring-8, he is also a visiting professor at Kobe University.

About SPring-8

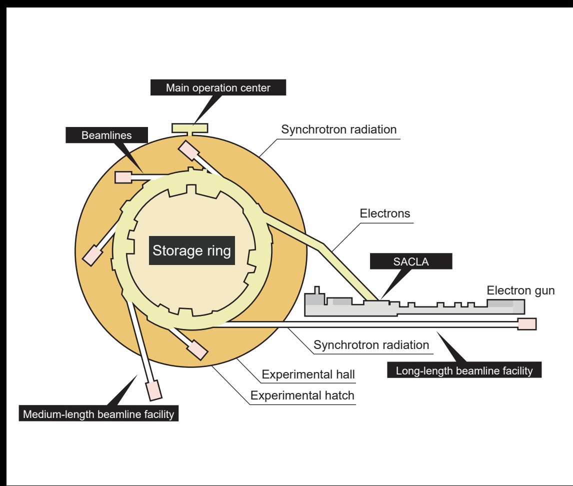
SPring-8, a world-class synchrotron radiation facility

Q The samples from the asteroid Ryugu were brought to SPring-8, where you work, Dr. Uesugi. Could you explain what kind of facility SPring-8 is and its significance in your research?

SPring-8 is a facility that generates synchrotron radiation by accelerating electrons and conducts experiments using the generated synchrotron radiation. Synchrotron radiation includes light of all wavelengths, and SPring-8 is equipped to produce X-rays of particularly high energy (i.e., short wavelengths). Synchrotron radiation X-rays produced from electron energy of 8 GeV (giga electron volts) have high flux density, high directionality, high brightness, and can irradiate a very small area. SPring-8 uses this light for the structural analysis of microscopic materials. Producing high-brightness synchrotron radiation requires a massive facility measuring about 500 meters in diameter.

SPring-8 consists of three main facilities: a facility to generate high-energy electron beams (currently, electrons generated at SACLA are partially used at SPring-8), a facility to keep the accelerated electron beams rotating at a constant speed (storage ring), and beamlines using synchrotron radiation. The higher the electron energy, the brighter the light with better directionality, and the greater the change in the direction of the electron beam, the more light of shorter wavelengths, such as X-rays, is included. In other words, to obtain large energy, the size of the accelerator must be increased. This is one of the reasons SPring-8 is one of the largest facilities in the world, and the synchrotron radiation it produces is among the best in the world.

SPring-8 is a circular radiation facility with a diameter of about 500 meters. It is so big that people sometimes ride bicycles around the storage ring building that houses the beamlines.



Internal structure of SPring-8

A specialist team for facilities and supporting users

Q What kind of institution is the Japan Synchrotron Radiation Research Institute (JASRI), where you work?

JASRI is a research institute that operates, maintains, supports the use of, and develops the necessary technologies for the SPring-8 large synchrotron radiation facility and the SACLA X-ray free-electron laser facility, which is operated by the National Institute of Physical and Chemical Research (RIKEN).

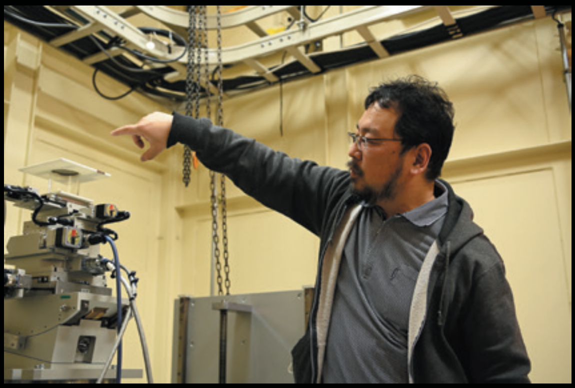
SPring-8 and SACLA are widely open to the public domestically and internationally. Many highly skilled staff members operate the world's most advanced synchrotron radiation facilities to ensure smooth, high-level use.

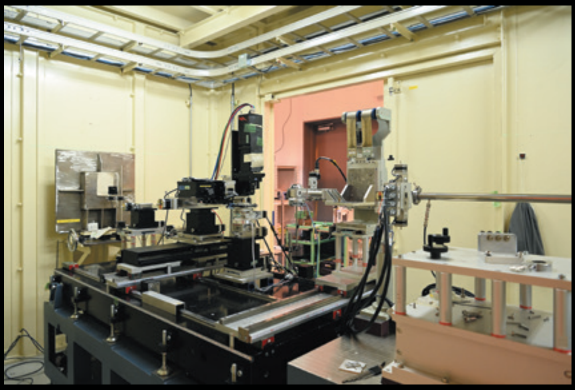
The research facilities are located in Harima Science Garden City in western Hyogo Prefecture. The vast site includes several experimental facilities and related facilities that support day-to-day research.

Analyzing the structure of materials using the synchrotron radiation X-ray

Q **Could you tell us why X-rays are used among the different wavelengths emitted by synchrotron radiation and what we can learn from experiments using X-rays?**

When you look at a sample with a visible light microscope, you start at a low magnification, say 5×. Once you have identified the area you want to observe, you can increase the magnification to 10×, 50×, etc., to focus on even finer areas. However, sometimes you cannot see what you want simply by turning the revolver of a microscope to change the magnification. You can then adjust the area and focus or adjust the amount of light entering the field of view since the narrower the field of view, the darker the field of view is. As I mentioned earlier, synchrotron radiation X-rays are much denser and can irradiate a much smaller area than X-rays produced by conventional X-ray sources. They are suitable for observing very fine objects such as the sample grains from Ryugu. Another notable property of X-rays is diffraction. Every material has a unique angle of diffraction of X-rays, so if you know the value of the angle of diffraction, you can determine the period of the material to which the X-rays were applied. In other words, after passing through the sample, the X-rays reveal the constituents of the sample.





Diffraction, along with imaging and spectroscopy, is a common method for determining the state of matter. In particular, diffraction is extremely effective at obtaining information by irradiating light on a tiny area. Now that we know just how useful X-ray synchrotron radiation is, let's build a large synchrotron radiation experimental facility to get information from even finer objects! And so, SPring-8 was born. Many new technologies that are advancing synchrotron radiation science have been developed at SPring-8. In the past, when people asked me, “What is SPring-8?” I often replied, “It's a big microscope,” but now I think I can just say it’s “an X-ray microscope.”

The Path to Ryugu Sample Analysis and Future Prospects

The beginning of a multi-year analysis project

Q Could you tell us how you got involved in the analysis of the Ryugu samples?

A total of five teams—the Japan Aerospace Exploration Agency (JAXA), the National Institute of Polar Research, the Institute for Molecular Science, and the Japan Agency for Marine-Earth Science and Technology (JAMSTEC), in addition to SPring-8—are involved in preparing the Ryugu samples for analysis. These five organizations signed an agreement and began their preparations in 2015. My involvement in the analysis project began when the SPring-8 team, of which I was a member, raised its hand to help with the analysis. From that point on, we spent about five years preparing for the sample analysis.

Five years of preparation, one year of sample analysis

Q Given the length of the preparation period, I assume there must have been numerous issues to address. What specific considerations did you take into account?

Our team was not only concerned with the analysis of the samples but also with other related issues. We carefully considered how to transfer the samples brought back from Ryugu to the tray in JAXA's clean chamber, how to divide the samples into small portions, and how to transport the samples to each facility. Since the sample analysis experiments are performed under vacuum or nitrogen conditions, we had to plan the experimental procedures, create the necessary instruments and containers, and develop new techniques for the experimental setups. In any case, we created everything from scratch through trial and error. Addressing these issues was a step-by-step process. Whenever someone came up with an idea, other members would suggest it might be useful for another purpose, and then they would make a prototype or do a test run. If it didn't work, another idea was considered. This was repeated over and over. It was time-consuming, but the collaborative team shared their wisdom across organizational boundaries to prepare for the sample analysis.

Ryugu taken from a distance of about 22 km
(Photo courtesy of JAXA, University of Tokyo, etc.)

In the preparation phase, I considered the design of a sample holder with carbon nanotubes and a container to transport the samples without exposing them to the atmosphere. Many of my ideas are reflected in their designs. This isn't exactly related to SPring-8, but JAXA has a curation facility where we planned to open the samples from Ryugu delivered to the ground, measure them with a spectrometer, measure their morphology, and take images. In order to do this, we needed a special sample holder and a device for relocation and storage. Curation team at JAXA came to me, saying "Mr. Uesugi, help us!" So, I took on the role of solving the problem. I was singled out or chosen from the collaborative team because, I guess, at that time they did not have the skills or knowledge to design holders and containers, and they were too busy to find the time to research it. So, that's how things happened.

You can only bring limited materials into the curation facility and need to work in a chamber with rubber gloves. So, we need to make the operation mechanism and dimensions as simple as possible. Since anyone may operate it, a mechanism that requires a lot of force on the fingertips should be avoided. While discussing the matter with JAXA staff, I determined the material and shape of the glass containers for holding the samples, and designed from scratch the multi-stacked, high-density storage system for storing these containers. The case of Ryugu required a lot of careful consideration with great care.
From there, while working with JAXA in a super rapid fashion, we drew drawings, made prototypes, and created prototypes with a 3D printer until we completed the project. Many of my designs are found in the chamber of the curation facility. In fact, these efforts have not earned me a single penny. I just did them as my hobby. But I took them on out of curiosity. This is how the sample holders and transport containers were made. It's a secret but I guess I am allowed to tell you.

Unforeseen challenges in analyzing Ryugu's samples

Q **Even with all this careful consideration, were there any unknown factors that only became apparent once you actually obtained the samples? If so, could you share what those were?**

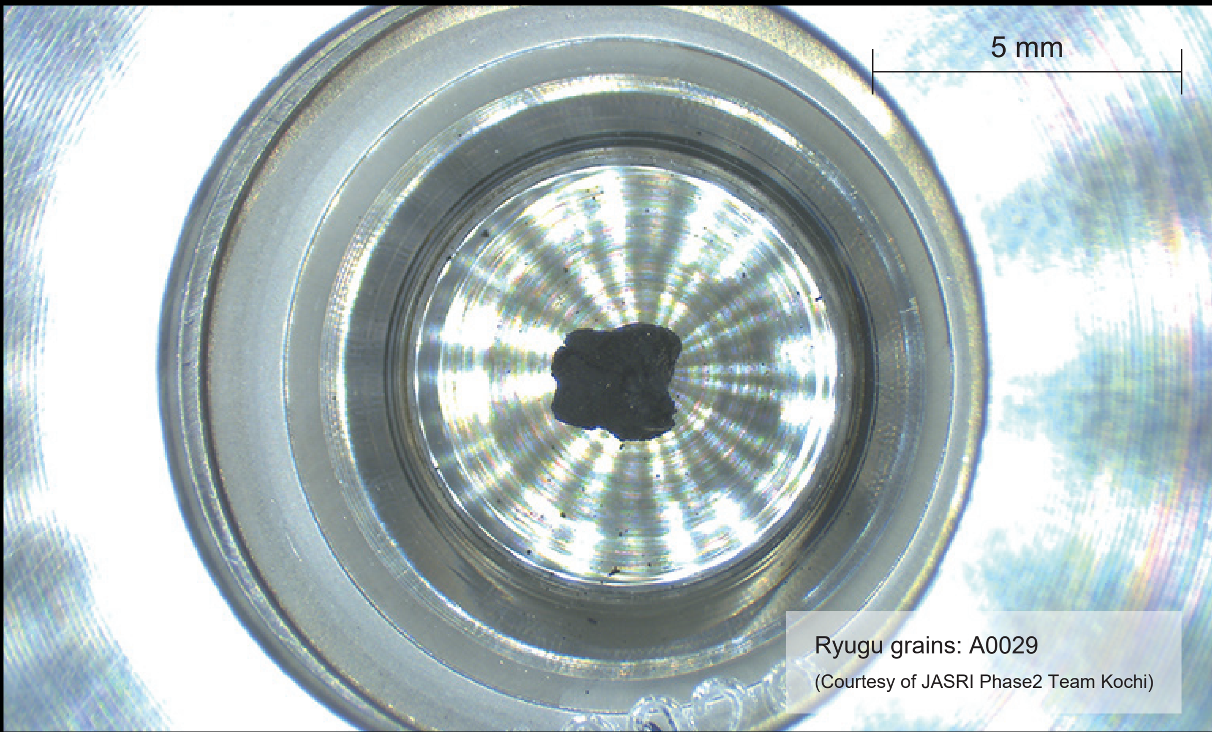
We already knew that Ryugu was a C-type asteroid. C-type asteroids appear black when observed. This was also known from analyzing meteorites that had fallen to Earth, which showed that black materials contain large amounts of organic compounds. The presence of organic compounds suggests that it has never been exposed to temperatures of 100 °C or 200 °C since the formation of our solar system. This is because exposure to temperatures above 100 °C would evaporate both the water and the organic compounds. Asteroids without water and organic compounds appear white when observed, which is said to be the case with an S-type asteroid. Since Ryugu looks black, we guessed that it is a C-type asteroid, and that the samples that would reach the ground would contain both water and organic compounds. However, we could not be certain whether the samples had not been contaminated with substances on Earth, whether they had not been exposed to water, or what they were like before Ryugu was formed. We could only guess to the extent of “it may be so.” The most difficult thing to predict was the hardness. At the preparation stage, we had no idea whether the samples would be hard or fragile.

Since we didn't know much about the samples, we had to be careful not to expose them to the atmosphere, to water and oxygen, and to avoid rolling them over. But when they returned to Earth, they would enter the atmosphere, the parachute would open, and boom! They would hit the ground with a significant impact, causing the samples to experience a shock. Since I expected some physical damage to the samples, I was very careful about the material of the sample holders to avoid contamination. JAXA had restrictions on the materials that could be used in their clean chambers, so we learned about these conditions and decided that materials such as quartz or sapphire should be used for glass parts and Viton only for rubber.

The historic moment of taking a close look at the samples after five years of dedication

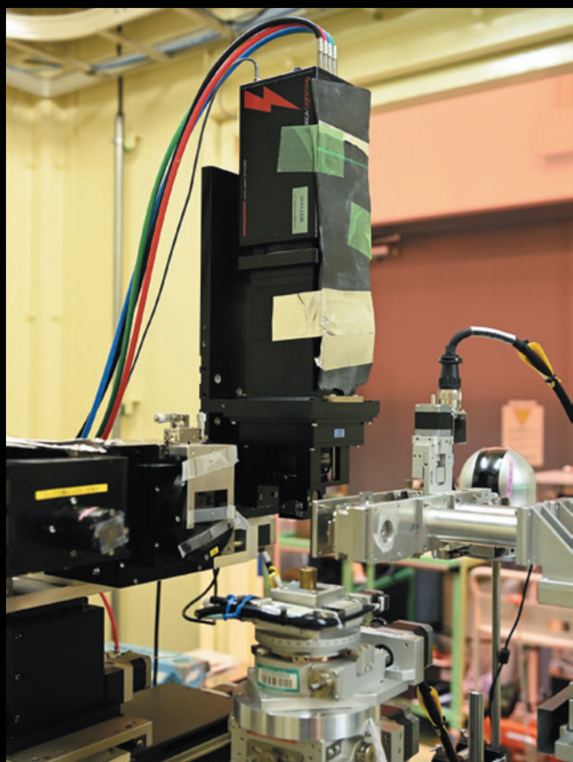
Q **What was everyone's reaction when the samples were transported to SPring-8 after safely landing on the ground?**

Once the samples were safely delivered and placed on the experimental apparatus at SPring-8, the team members began competing to photograph them. With the lab barely able to accommodate two or three people, more than ten team members took turns entering the lab to capture the historic moment on camera.



The size of the samples from the asteroid Ryugu ranged from 3 mm to 10 mm. They were placed on an experimental apparatus and their tomograms were taken with an X-ray CT. The images were projected on a large monitor in an adjacent room so that a large number of people could observe them together. Several samples were provided, so we took images and discussed them while projecting the results one by one. Every time an image appeared on the screen, voices rose from the audience with exclamations like “Wow!” or “Oh, it's cracked!” or “What in the world is that clump?” Inferential comments from a meteorite expert present at the meeting fueled even more lively discussion. We also had online meetings with officials in the UK.

The actual imaging showed that some samples were as expected and some were unexpected. I was impressed by the unexpectedly smooth texture, or rather, the uniform appearance. We used Hamamatsu Photonics’ ORCA®-Lightning for the CT imaging. It worked well for this application.



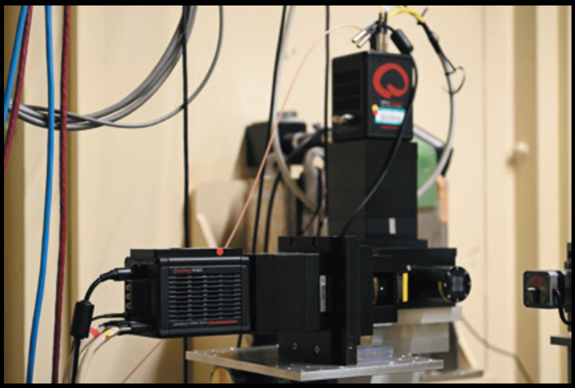
The ORCA-Lightning used for Ryugu’s sample analysis

Traces of water presence found on Ryugu

Q What have you learned along the way from your X-ray CT analysis?

A closer look at the X-ray CT image reveals the presence of a carbonate mineral ($CaCO_3$), well known as calcite and aragonite. Carbonate minerals are substances that cannot be formed without water. Their existence means that Ryugu had water or was in an environment where it was exposed to water. There were many clumps of various sizes. Why are there so many clumps, and why are they of different sizes? Our members considered whether the presence of multiple clumps indicated at least two exposures to water. Were there at least two periods of exposure to water, one when small clumps formed and another when large clumps formed? These findings were obtained over several days of viewing CT images.
The samples also contained a lot of clay minerals in addition to carbonate minerals. If you imagine the soil in a schoolyard, you can better understand that water is needed to make clay minerals, and the presence of clay minerals proves that there is interlayer water. It is not yet known how much water was present, but it is evidence that water was present.

This was only a small part of what we discovered, and through repeated experiments at SPring-8, we determined that there are three patterns of evidence for the existence of water: a substance that has been exposed to water, a substance that contains water, and a substance that is itself water. It is very difficult to analyze Ryugu because it contains iron and other minerals in addition to water and the aforementioned minerals. Research teams outside of SPring-8 are also involved in the analyses, and I am sure that many more facts will be clarified in the future.

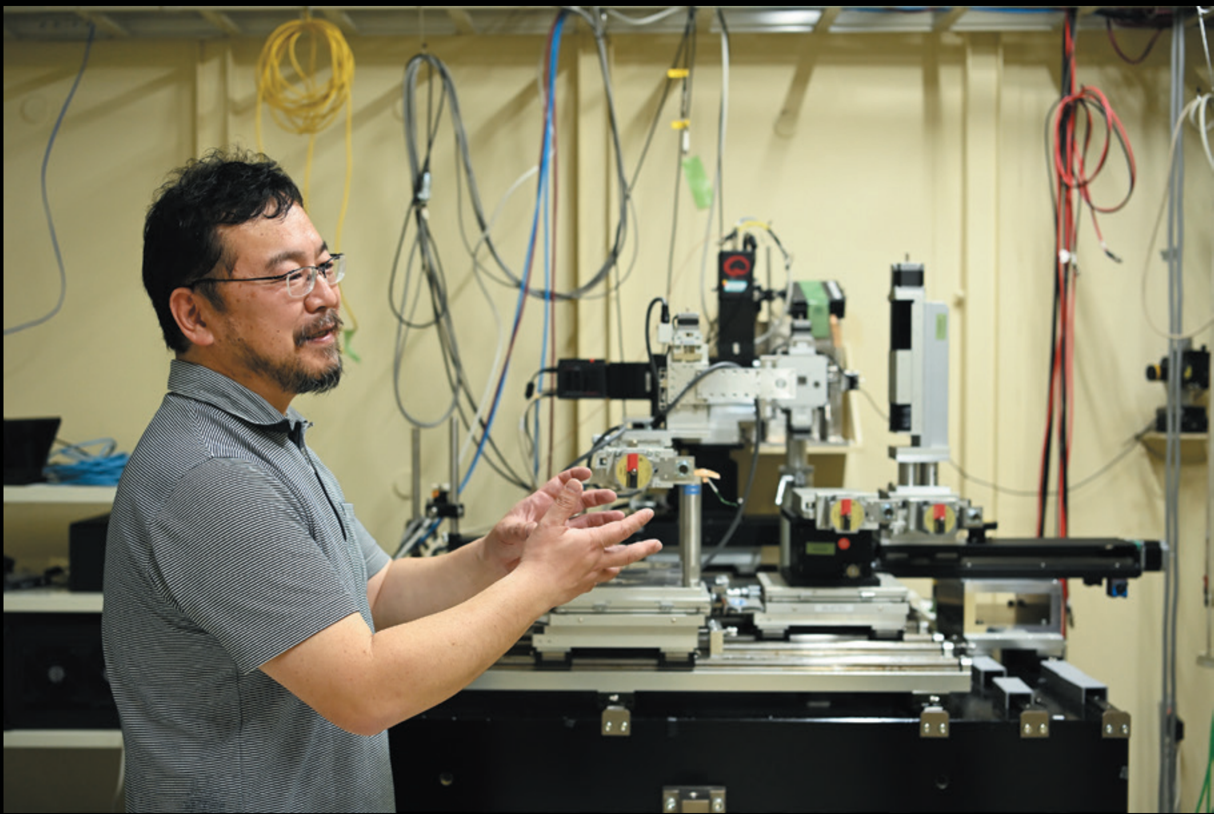


Discovering one clue at a time to solve the mysteries of the origin and evolution of our solar system and the birth of life

Q How will the results of the Ryugu sample analysis contribute to the development of life sciences? What are your expectations?

I cannot say whether the findings will lead to the development of life sciences because I am not an expert, but what we have learned in the process of analyzing Ryugu is that there was a kind of precursor to Ryugu. We call it the parent body, which seems to have existed much farther away than Jupiter. In the past, our solar system was made up of many small celestial bodies that collided with each other. The dust from the collision drifted around and collided again, and so on. It is said that Mercury, Venus, Earth, Mars, and Jupiter, which are in the present solar system, were formed by the gradual growth of older bodies being incorporated into new bodies. Ryugu's parent body was destroyed by a similar process and part of it became a rubble pile satellite called Ryugu. The sun's gravitational pull then pulled the asteroid toward the sun, causing it to orbit near the Earth. This is the present story of Ryugu, of how it came to be. If we take a comprehensive view, using not only non-destructive analysis, which I do, but also destructive analysis, chemical analysis, etc., we will be able to depict the life of Ryugu from its birth to its disappearance, like the asteroid Itokawa, which is already known to disappear in another 1 billion years. I believe what we can learn from depicting the life of the asteroid is that we will be able to understand what happened before the current solar system was formed, that is, the origin and evolution of our solar system.

Even when you see a video at a museum explaining the origin of our solar system, you see a primitive solar nebula spinning around, and before you know it, the current solar system appears out of nowhere, right? This is because no one knows the exact origin or the process of its evolution. I think the process is really interesting, but it is very difficult to learn about. The only way to solve the mystery is to gather evidence from samples successfully returned from celestial bodies such as Ryugu, Bennu, and the Martian satellite Phobos. As we gather information, we will be able to unravel, one by one, what we did not know, and develop a common view of the history of the solar system that can be properly explained. In this way, I believe we can shed light on the origin of life. I hope to know some exciting results, maybe in 20 years or so. I will be about 70 years old then, hopefully still alive.



Hamamatsu Photonics' user-centric design and accurate data presentation

Q **Dr. Uesugi, you have been using our products, mainly cameras, for many years. We would love to hear from you about the appeal and benefits of using our cameras.**

First of all, the measurement data is properly displayed accurately and without unnecessary adjustments. It is important to the user that unnecessary adjustments are not made to the image through image processing, etc. Second, I like the way the camera and software are designed from the user's perspective. They seem to be crafted based on the type of data users need. I also think that the engineers who develop these cameras must be quite passionate about their work. We ask many of your engineers to help us achieve our goals, and they consistently provide the answers we expect. I hope that Hamamatsu Photonics will continue to innovate and produce unique products that only your team can create.

Product lineup

High resolution X-ray imaging system AA51

M11427-57B, -57S, -58B, -58S

By simply selecting the main body of the X-ray imaging system, the phosphor screen*, and the optical system*, you can easily acquire X-ray images. You can freely choose a camera from the lineup and build a system by combining the optical system and camera according to your application.

Additionally, the optical design considers the durability and maintainability of the device, making it suitable for imaging using strong X-rays used in synchrotron radiation facilities.

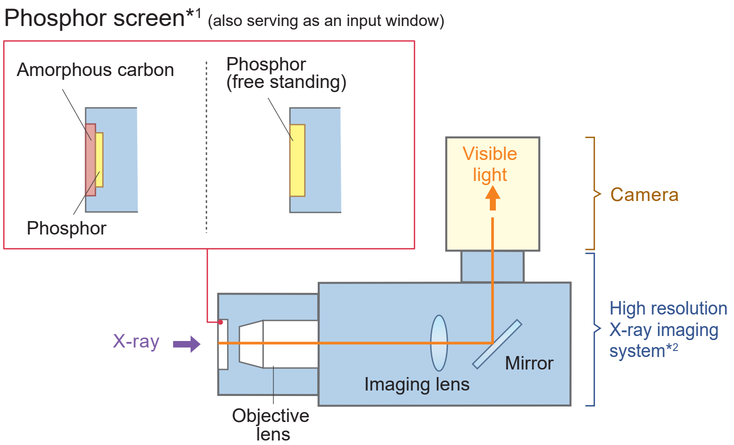
*Options

Specifications

Product number	M11427-57B, -57S	M11427-58B, -58S
X-ray energy	6 keV or higher	
Phosphor effective diameter	Refer to specifications for phosphor screens (below)	
Phosphor material		
Peak emission wavelength		
Decay time		
Thickness of phosphor (typ.)		
Base material of phosphor		
Spatial resolution *	1 μm or less	800 nm or less
1st lens	10× (NA 0.45)	20× (NA 0.75)
2nd lens	200 mm	

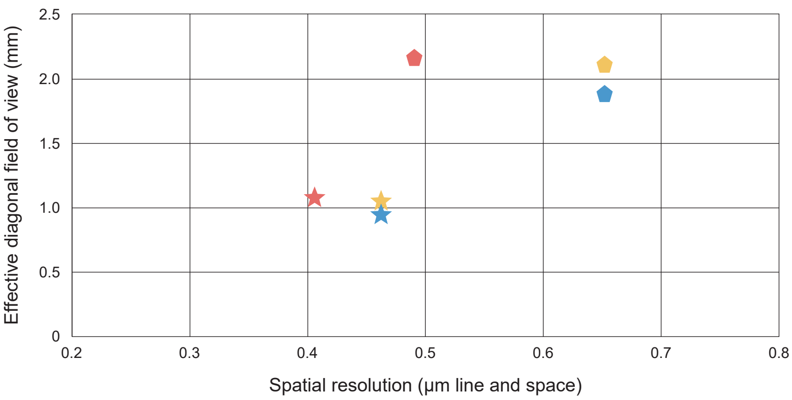
* Reference value with ORCA-Flash4.0 V3. It varies depending on the system configuration.

Light path



*1 Phosphor screen is an option.
*2 For the components of the imaging optical system, browning may be caused due to X-ray irradiation, resulting in a decrease in transmittance.

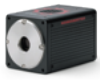
Correlation diagram of spatial resolution and effective field of view (reference data)*



	Imaging unit	Camera
Pentagon (red)	M11427-57 AA51	ORCA-Quest
Pentagon (blue)		ORCA-Flash4.0 V3
Pentagon (yellow)		ORCA-Fusion
Star (red)	M11427-58 AA51	ORCA-Quest
Star (blue)		ORCA-Flash4.0 V3
Star (yellow)		ORCA-Fusion

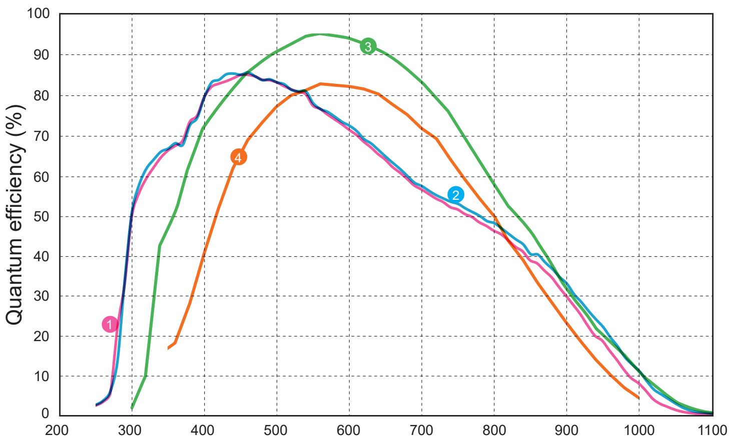
* The spatial resolution and effective field of view in the above diagram are examples of actual values measured with visible light without a phosphor. Please refer to it as reference data.
Please contact Hamamatsu for detailed measurement conditions.

Option Camera

Camera	ORCA-Quest 2 qCMOS® camera	ORCA-Fire Digital CMOS camera	ORCA-Fusion BT Digital CMOS camera	ORCA-Flash4.0 V3 Digital CMOS camera
Product number	C15550-22UP	C16240-20UP	C15440-20UP	C13440-20CU
				
Sensitivity wavelength range (nm)	1	2	3	4
Effective number of pixels (H×V)	4096 × 2304	4432 × 2368	2304 × 2304	2048 × 2048
Pixel size [μm (H) × μm (V)]	4.6 × 4.6	4.6 × 4.6	6.5 × 6.5	6.5 × 6.5
Effective area [μm (H) × μm (V)]	18.841 × 10.598	20.387 × 10.892	14.976 × 14.976	13.312 × 13.312
Full well capacity (electrons, typ.) *1	7000	20 000	15 000	30 000
Readout speed (frames/s, typ.) *1	Standard scan 120 Ultra quiet scan 25.4 –	Full resolution 115 Vertical 4 line 19 500 –	Fast scan 89.1 Standard scan 23.2 Ultra quiet scan 5.42	Standard scan 100 Slow scan 30 –
Readout noise (electrons, rms, typ.) *1	Standard scan 0.43 Ultra quiet scan 0.30 –	Full resolution 1.0 – –	Fast scan 1.6 Standard scan 1.0 Ultra quiet scan 0.7	Standard scan 1.6 Slow scan 1.4 –

*1 It varies depending on the conditions. Please contact Hamamatsu for details.

Spectral response



Option Phosphor specifications

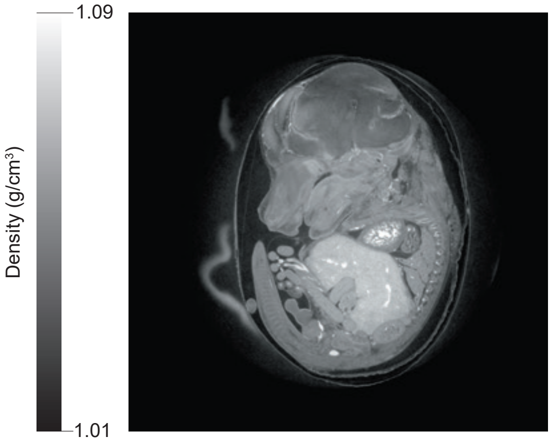
Bonding method	Product number	Phosphor material	Peak emission wavelength	Decay time	Phosphor thickness	Phosphor diameter	Phosphor effective diameter	Base material of phosphor	Space ring
Direct bonding	A15150-LU010DB	LuAG *1 $(Lu_3Al_5O_{12}: Ce+)$	535 nm	70 ns	10 μm	15 mm	10 mm	Amorphous carbon Diameter: 20 mm Thickness: 1 mm	Black plastic Outer diameter: 20 mm Inner diameter: 16 mm Thickness: 2 mm
	A15150-LU050DB				50 μm				
	A15150-LU100DB				100 μm				
	A15150-GA010DB	GAGG *1 $(Gd_3Al_2Ga_3O_{12}: Ce+)$	520 nm	92 ns	10 μm				
	A15150-GA050DB				50 μm				
	A15150-GA100DB				100 μm				
Glue bonding	A15150-LU010GB	LuAG *1 $(Lu_3Al_5O_{12}: Ce+)$	535 nm	70 ns	10 μm				
	A15150-LU050GB				50 μm				
	A15150-LU100GB				100 μm				
	A15150-GA010GB	GAGG *1 $(Gd_3Al_2Ga_3O_{12}: Ce+)$	520 nm	92 ns	10 μm				
	A15150-GA050GB				50 μm				
	A15150-GA100GB				100 μm				
Free standing*2	A15141-LU	LuAG *1 $(Lu_3Al_5O_{12}: Ce+)$	535 nm	70 ns	1000 μm	20 mm	16 mm	–	
	A15141-GA	GAGG *1 $(Gd_3Al_2Ga_3O_{12}: Ce+)$	520 nm	92 ns					

*1 For LuAG and GAGG, a streak and white spots may occur. These are due to the characteristics of the single-crystal phosphor and are not a defect.
*2 It is necessary to block the ambient visible light in the operating environment.

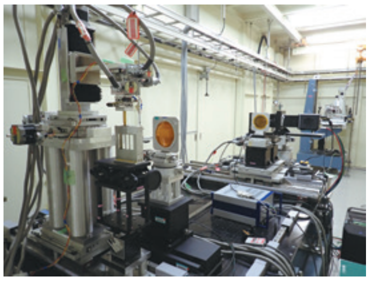
Applications

HEP / Synchrotron

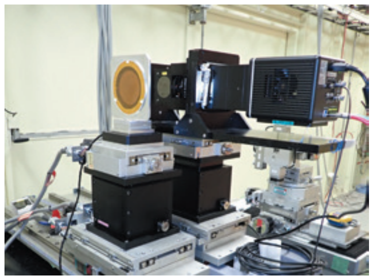
For imaging of X-ray or other kinds of high energy particles, a scientific camera coupled with a scintillator is often used. The imaging system must have low noise and high speed to detect momentary phenomena.



Experimental setup

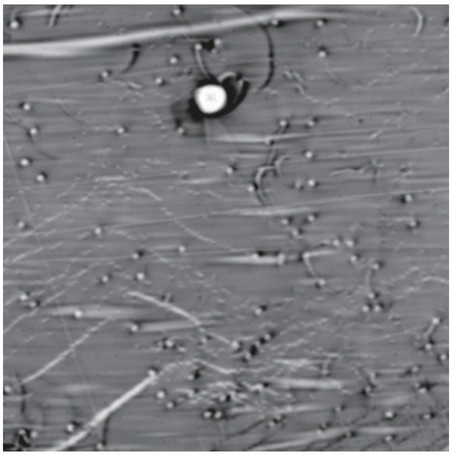


Camera setup



- X-ray phase contrast CT image of a mouse embryo from an ORCA-Quest combined with high resolution X-ray imaging system (M11427)
- Exposure time: 15 ms, Total measurement time: 6.5 min
- Data courtesy of: SPring-8 BL20B2 beamline by Dr. Masato Hoshino, Senior researcher in Japan Synchrotron Radiation Research Institute (JASRI)

SiC defect observation

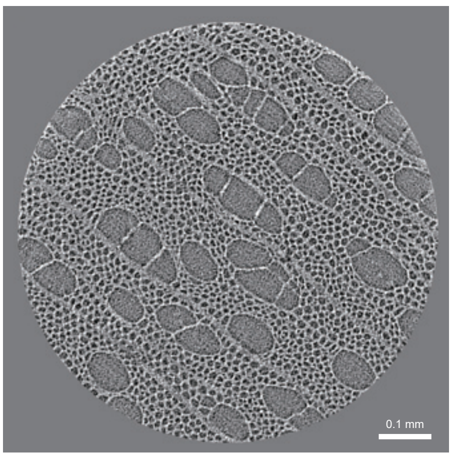


Test conditions

Method	X-ray topography
Camera	ORCA-Flash4.0 V3
Sample	SiC single crystal substrate (dislocations in the crystal)
X-ray energy	9 keV
Pixel size	0.65 µm
Magnification	×10
Exposure time	10 s
Number of pixels in the target image	1970 pixels × 1970 pixels (1.28 mm × 1.28 mm)
Phosphor screens	LuAG 10 µm

Data courtesy of:
Innovation Center for Semiconductor and Digital Future, Mie University
Yongzhao Yao, Ph.D

Internal observation of wood



Cross-sectional view after 3D image reconstruction

Test conditions

Method	X-ray CT
Camera	ORCA-Fusion BT
Sample	Toothpick
Exposure time	100 ms/projection
Number of projections	1800
X-ray energy	15 keV
Pixel size	0.65 µm

Data courtesy of: Photon Science Innovation Center

Related products

ORCA-Quest 2 qCMOS camera

C15550-22UP

Realization of photon number resolving by low-readout noise

The ORCA-Quest 2 is a camera that leverages the design technology cultivated at Hamamatsu Photonics to achieve an ultra-low noise performance of 0.30 electrons rms and high-speed readout.

Features

- Readout noise: 0.3 electrons rms*1
- Readout speed: 25.4 frames/s*1
- Dark current: 0.006 electrons/pixel/s (-35 °C)
- Effective number of pixels: 4096 (H)× 2304 (V)
- Realization of photon number resolving (PNR) output

*1 Ultra quiet scan

URL https://www.hamamatsu.com/all/en/product/cameras/qcmos-cameras/C15550-22UP.html

X-ray sCMOS camera

C12849-111U

A high resolution and high sensitivity X-ray sCMOS camera

The C12849-111U is a high resolution and high sensitivity X-ray sCMOS camera. The camera is suitable for micro object by achieving 33 lp/mm high resolution image. Also, the product is compact, making it suitable for use as an embedded device in Micro CT/Nano CT systems.

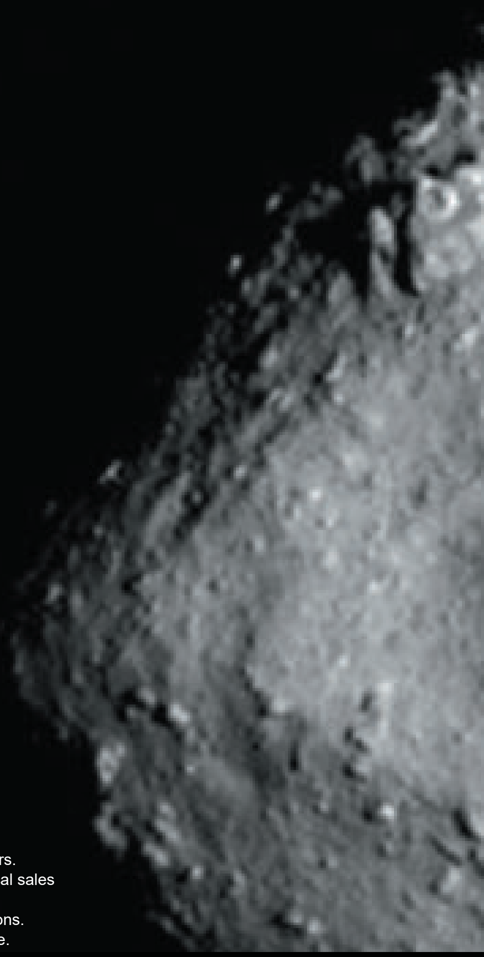
Features

- Resolution: 33 lp/mm
- Effective number of pixels: 2048 (H)×2048 (V)
- Readout speed: 30 frames/s

URL https://www.hamamatsu.com/all/en/product/cameras/x-ray-cmos-cameras/C12849-111U.html

Application & Case study

https://camera.hamamatsu.com/all/en/application_and_case_study.html



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