Application Notes

Double Tilt LN_2 Atoms Defend Holder

For JEOL

Product Summary

Holder

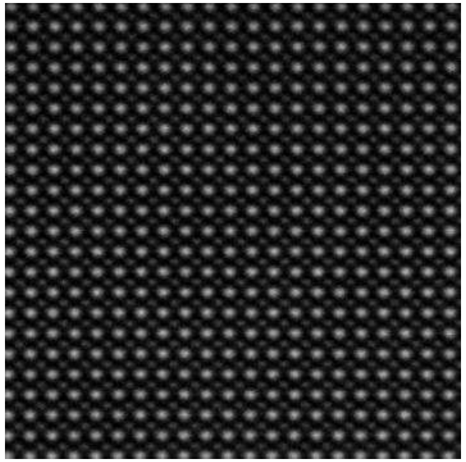
Basic Item(Recommended package)

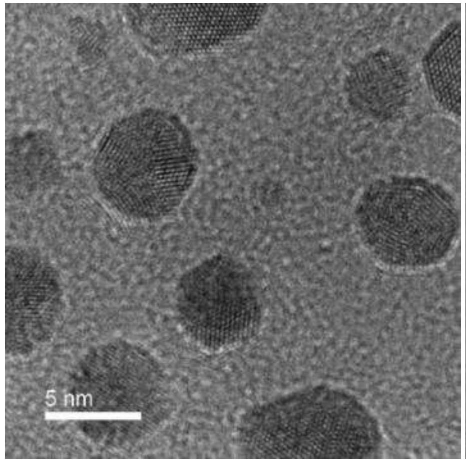
*Additional options are available

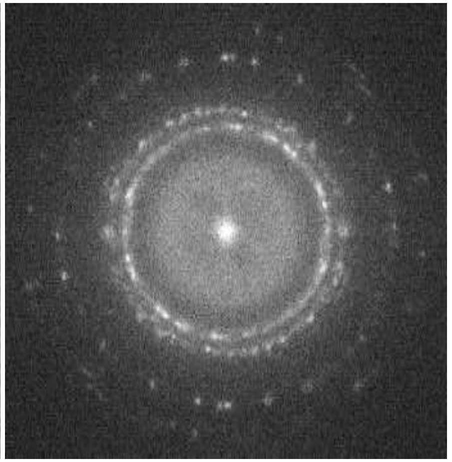
Temp. control unit	Gonio Cover	Safety Operation Stand	Torque Driver
			
safety standard	With Gonio cover Get the clear image	Easy and safety sample mounting in glove box	Manages the torque of screw fixing. Type:10mN

Specification

●Mechanism	:	**Double Tilt / LN_2 Cooling / Vacuum Transfer**
●Ultimate temp.	:	Guarantee temp.-160℃(-165±5℃)
●Arrival time	:	60 min
●X tilt	:	± 20 degree @ HR PP
●Y tilt	:	± 20 degree @ HR PP
●Resolution	:	0.24nm Au Nano Particle TEM FFT 002 observation (Factory check specification)
●Transfer	:	Ultimate vacuum inside the storage <1X10-3Pa
●Drift Rate	:	6 nm/min (to be improved)





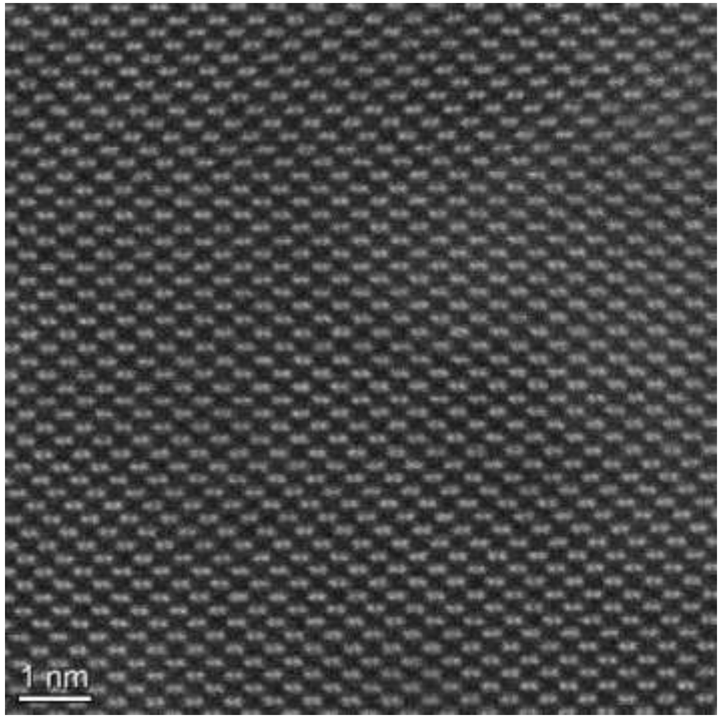


Mel-Build

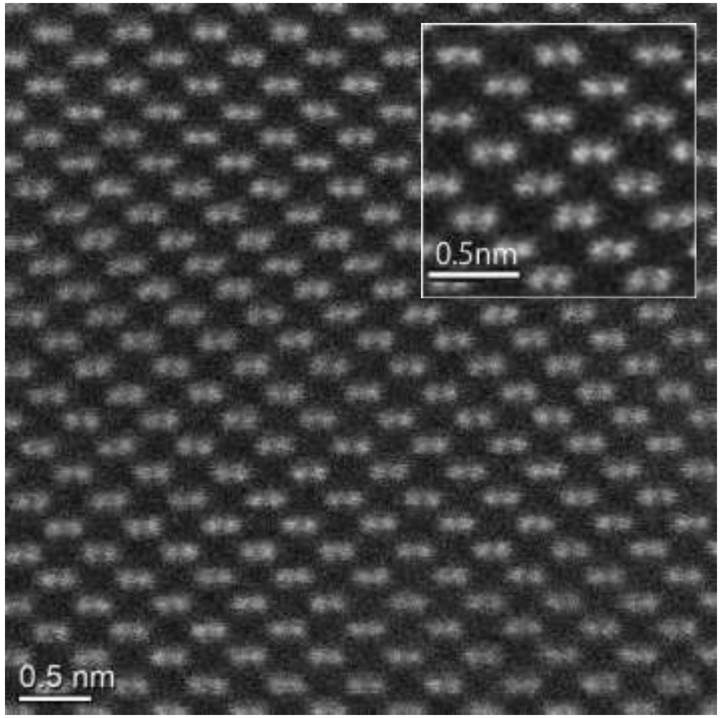
Latest Atomic Resolution Data

Atomic resolution observation of Si substrates after temperature reached in -170 ℃ (90 min).

Observation at 20x



Observation at 40x



Test summary

●**Sample**	:	Si substrates
●**Original Gonio cover**	:	In use
●**Photo Conditions**	:	1024×1024pixel
●**Pixel time**	:	5μsec（20M）、3μsec（40M）
●**Vacuum level**	:	2×10^{-5}Pa

Results

●**RT**	:	X +36.8um, Y -76.1um
●**Cooling**	:	X +76.8um, Y -68.7um
●**Misalignment**	:	X 40.7um,Y 7.4um

Provision of data

Kobelco Research Institute, Inc.

A good quality silicon structure was observed.

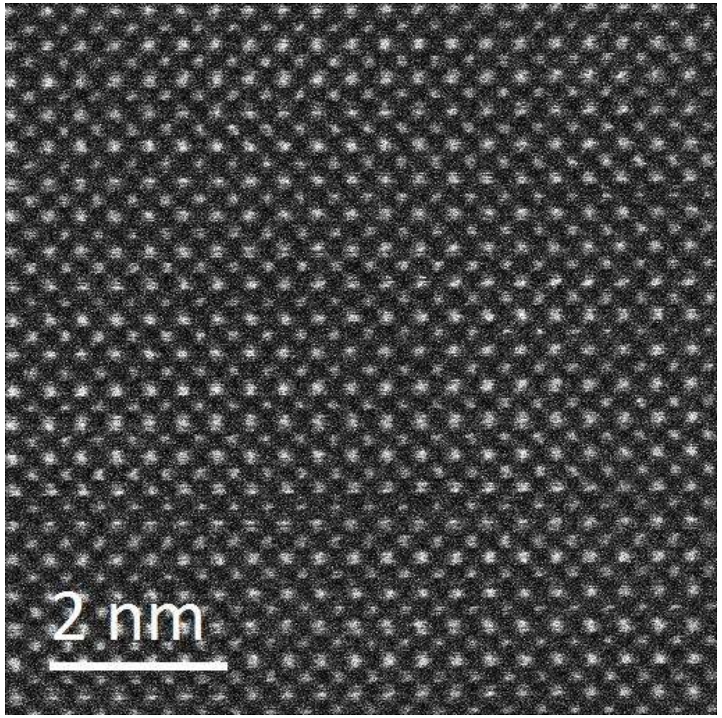
*not champion data.

*If the laboratory environment is improved further, better data may be available.

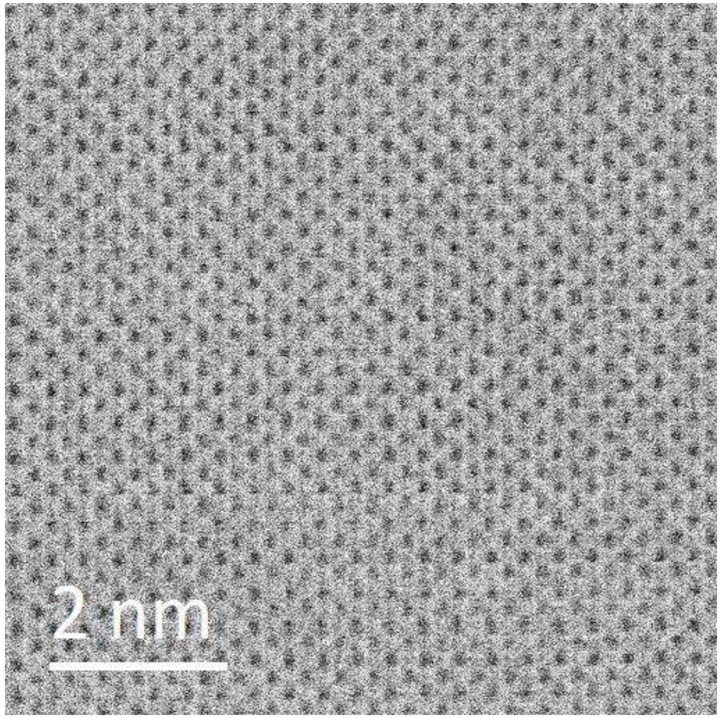
HR-STEM observation

Achieving HR-STEM observation at a magnification of 50M at -160 ℃

HAADF



BF



Test summary

●**Sample**	:	$SrTiO_3$
●**Magnification**	:	STEM 25 M
●**Equipment**	:	ARM 200F
●**Start time**	:	After reached -169 ℃

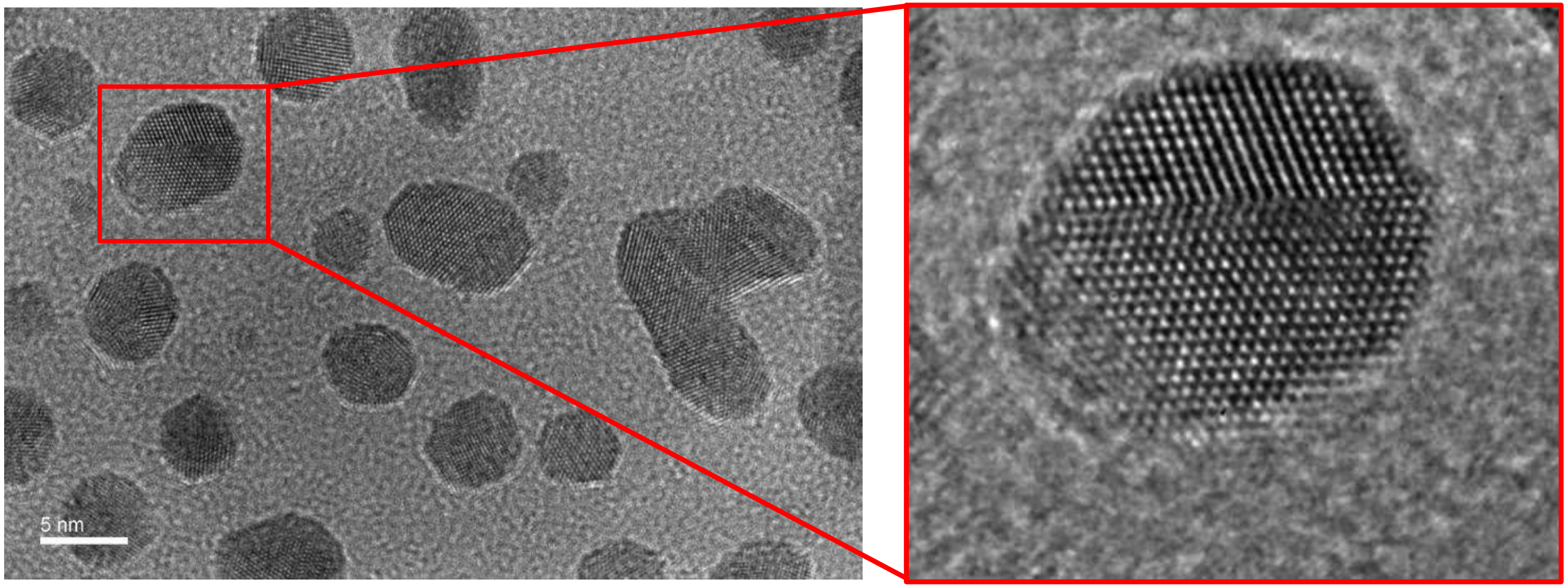
Provision of data

Dr. Kumamoto, Dr. Nakayama and Dr. Ishikawa,
Prof. Shibata Prof. Ikuhara

University of Tokyo.

HR-TEM observation

HR-TEM observation was performed immediately after temperature reached in -170 ℃ (60 min).

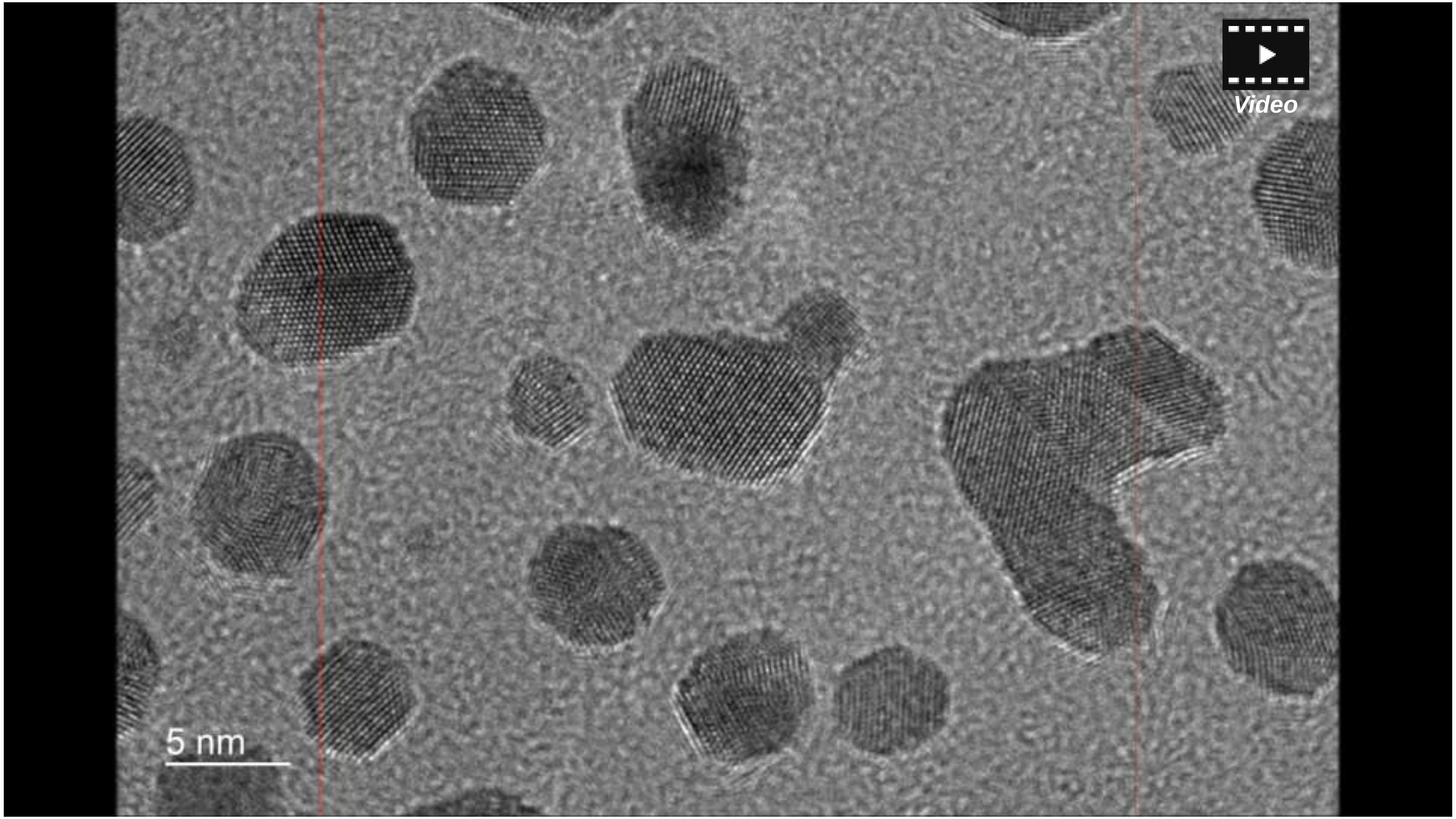


Test summary

●**Start time**	:	After reached -170℃
●**Sample**	:	Au nanoparticles on Cross grating
●**TEM**	:	ARM-200F

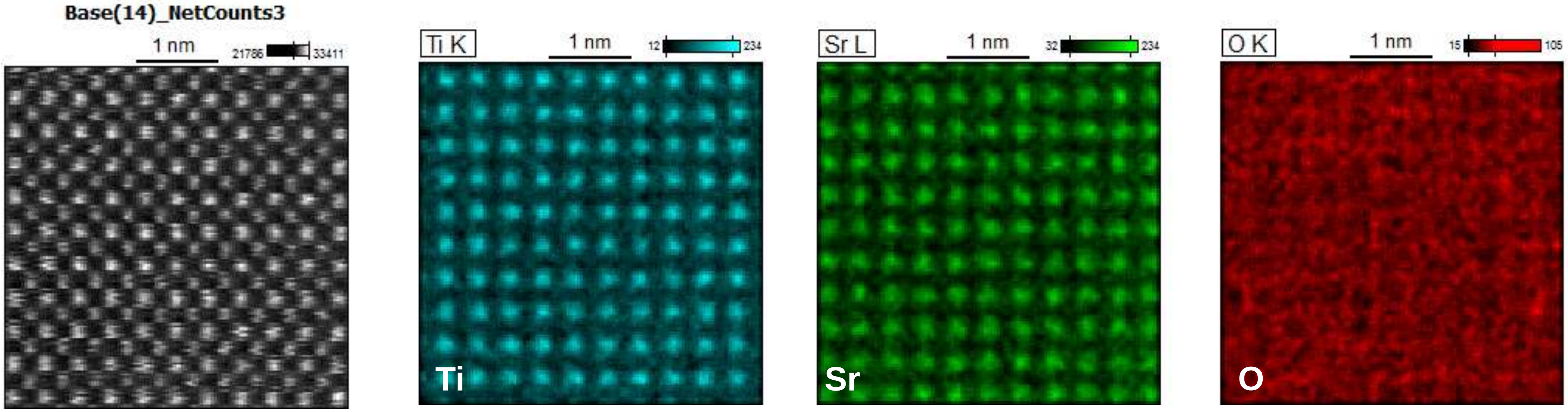
HR-TEM observation

A HR-TEM movie after lapse of about 30 minutes after reached -170℃ Video

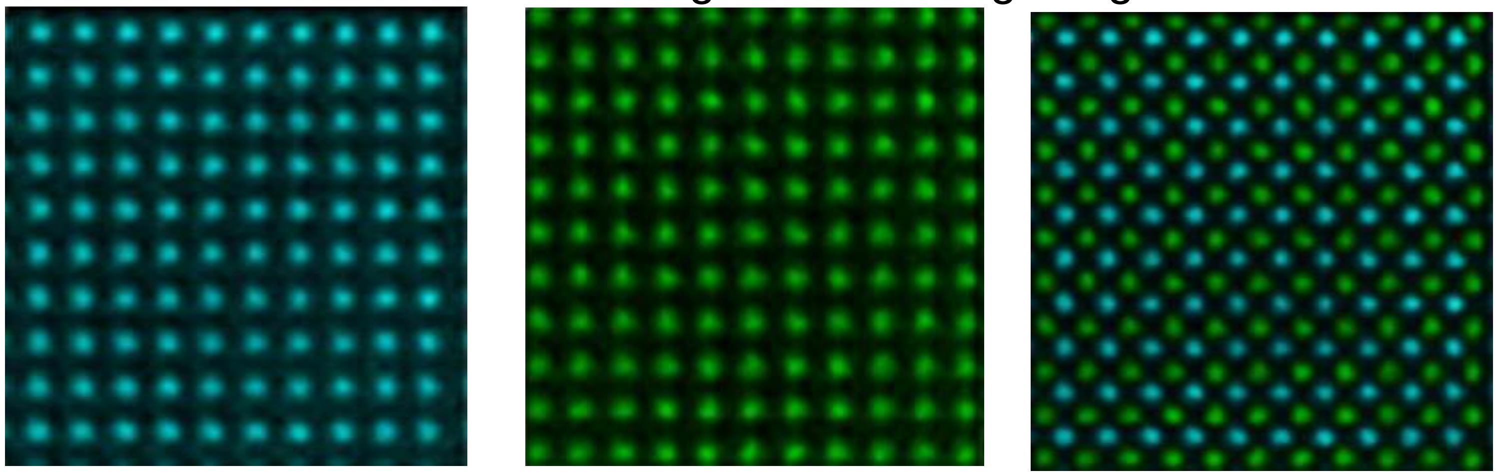


EDS analytical performance test

Using LN_2 cooling holder :Column mapping of $SrTiO_3$



STO After filtering and stacking image



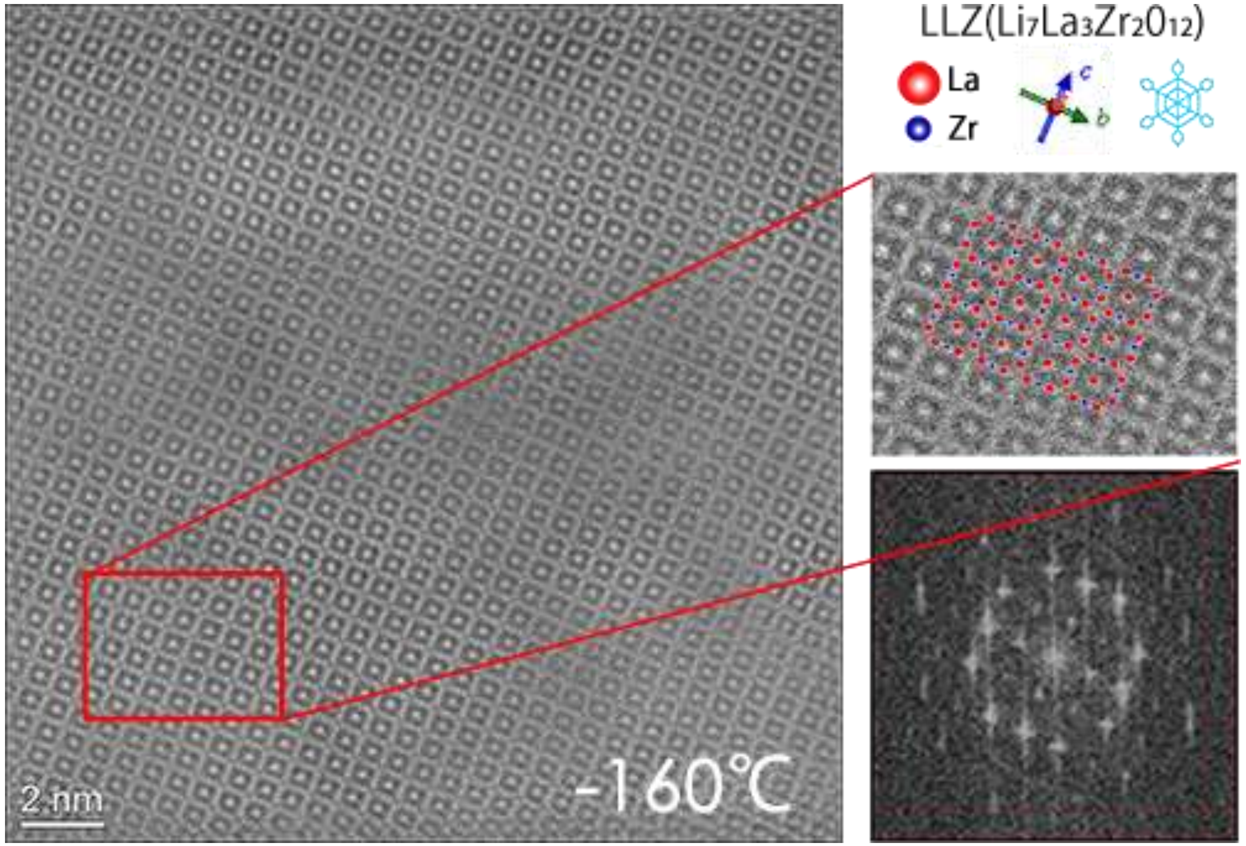
Provision of data

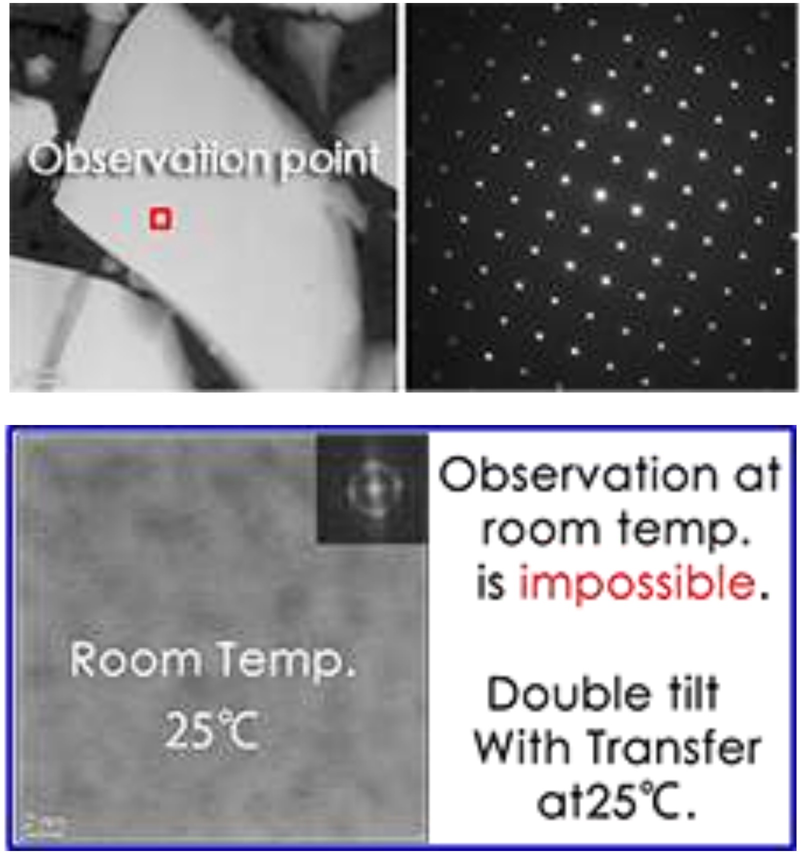
Dr. Kumamoto, Dr. Nakayama and Dr. Ishikawa,
Prof. Shibata Prof. Ikuhara

University of Tokyo.

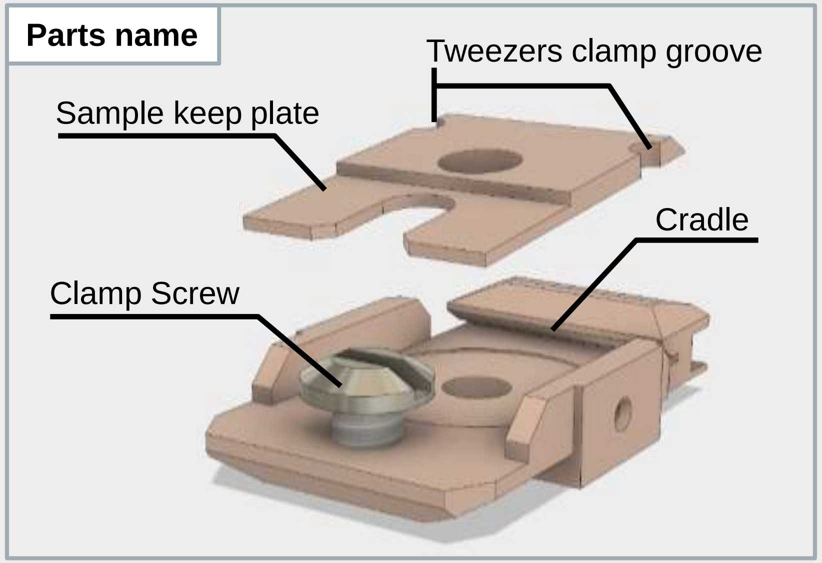
Atomic Resolution Test Summary

Atomic resolution STEM image observation of LLZ($Li_7La_3Zr_2O_{12}$),
which is degraded by exposure to air(moisture) and weak to electron beam with lithium ion material, was successful.From this result, it was proved that the non-exposure(Transfer) was successful, and the crystal orientation was adjusted by Y tilt,and the electron beam damage was reduced by cooling of -160℃.

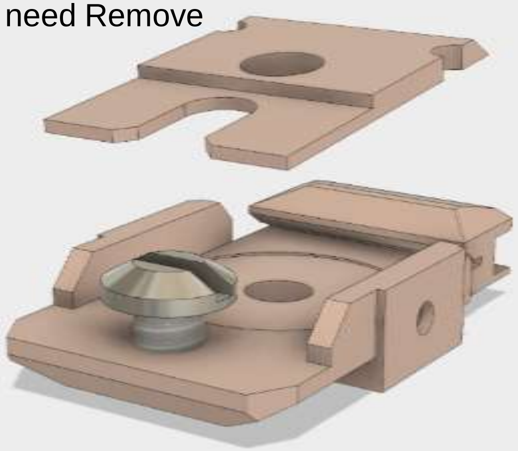




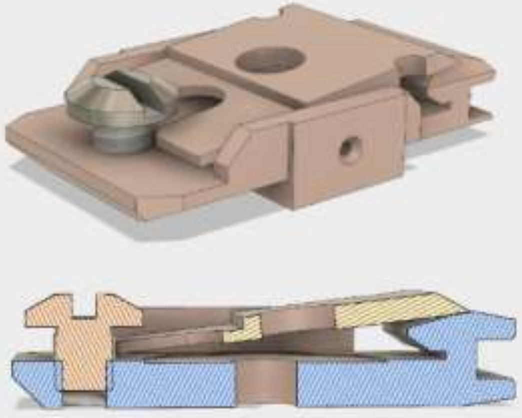
Sample set method



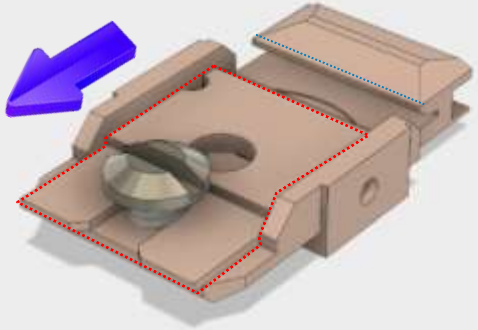
STEP1.
Loosen the clamp screw
No need Remove



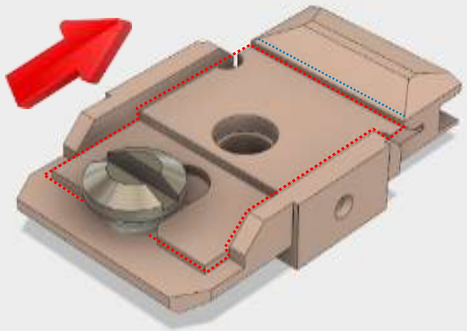
STEP2.
Place Sample keep Plate as shown below.



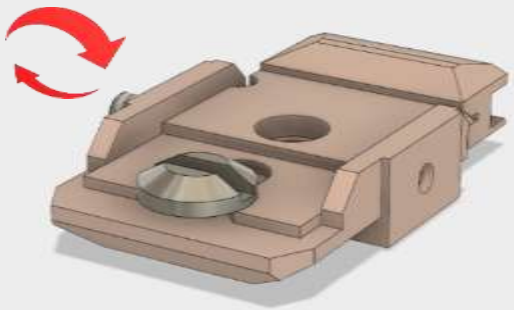
STEP3.
Use tweezers clamp groove to
slide in direction of blue arrow



STEP4.
Use tweezers clamp groove to
slide in direction of red arrow



STEP5.
Tighten the clamp screws



Gonio cover

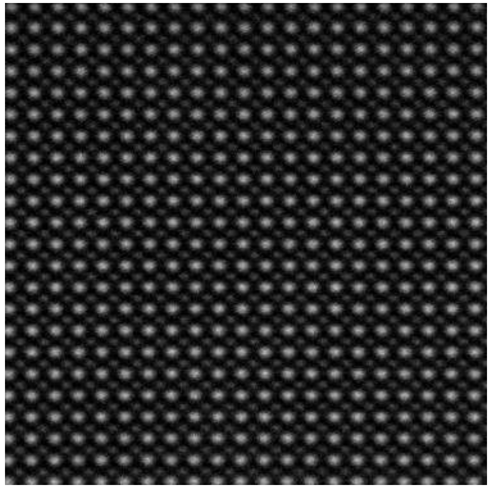
Temp. control with Gonio cover= grate resolution under -170C

Special Temp. control unit ±0.005℃

safety standard

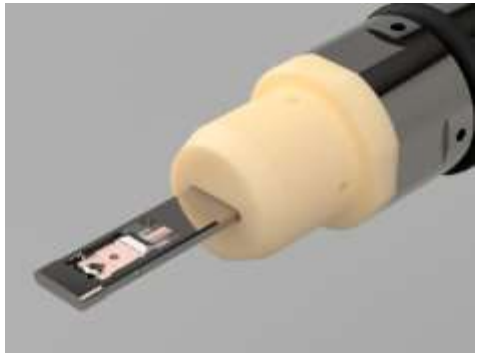
Original special Gonio cover.

Sample:BTO



Our holder and Temp. Control with Gonio cover Get the clear image

Maximum tilt angle

Holder Type	Figure	Available Pole-piece type	Holder Type Selection on Mel-Build software	Maximum tilt angle
Std. (WG/HR/FHP)		WGP HR FHP (*can't be used on UHR)	HR HR FHP -	X:>±35° Y: ±20° X:>±20° Y: ±20° X:>±15° Y: ±15° -
UHR		WGP HR FHP UHR	UHR UHR UHR UHR	X: >±35° Y: ±7° X: >±25° Y: ±7° X: >±15° Y: ±7° ↳(*unexamined) X: >±15° Y: ±7°

Mel-Build

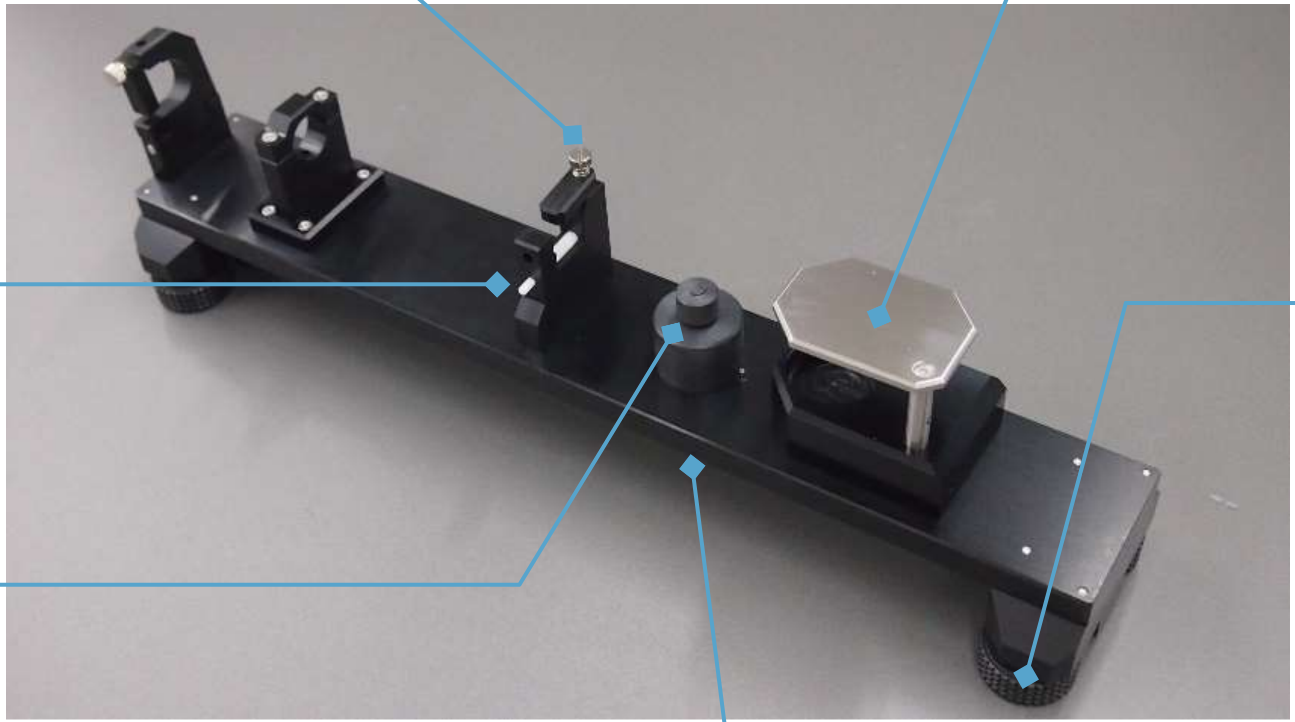
Safety Operation Stand

Working inside the glove box is very difficult,
A stable workbench is required when setting the sample

Holder holding plate:
Since you firmly clamp the holder, you can work safely until the work is completed.

Release button:
You can easily remove the holder from the pedestal with the release button

Mini jack:
We hold the specimen pedestal from below, weak strength, so you can fix the specimen safely. (Fitting sample holder ring from above, necessary for this proposal)

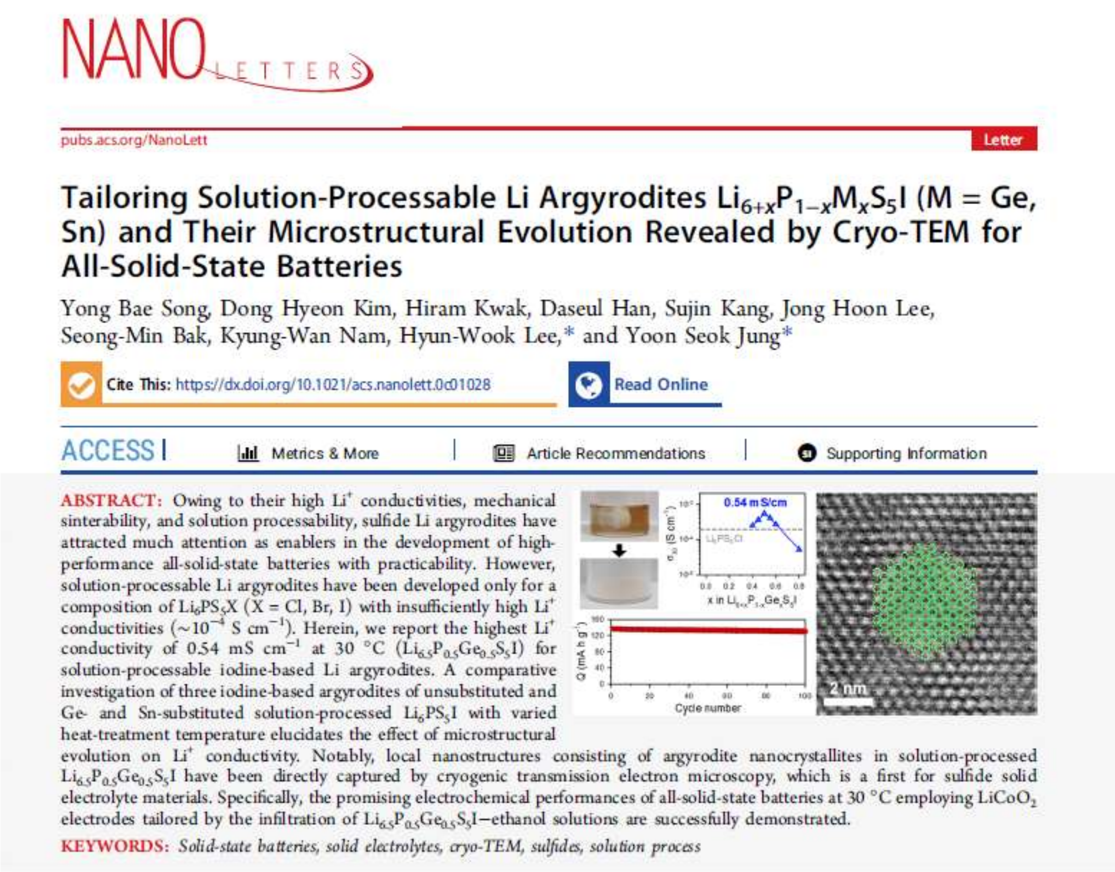


Palm rest:
You can rotate 360°, you can rotate it to your favorite position, when handling the sample when installing the sample.
Since the hand is fixed, you can attach the sample in a stable condition.

Height adjustment leg:
Since the height can be independently adjusted in 4 places, it can be adjusted so that stable work can be performed even with a slight distortion and inclination.

Bridge structure:
Since it has a bridge structure, it can work directly under an optical microscope.
(Some lights can not be used)

Publication of recent papers-1



NANO LETTERS

pubs.acs.org/NanoLett — Letter

Tailoring Solution-Processable Li Argyrodites $Li_{6+x}P_{1-x}M_xS_5I$ (M = Ge, Sn) and Their Microstructural Evolution Revealed by Cryo-TEM for All-Solid-State Batteries

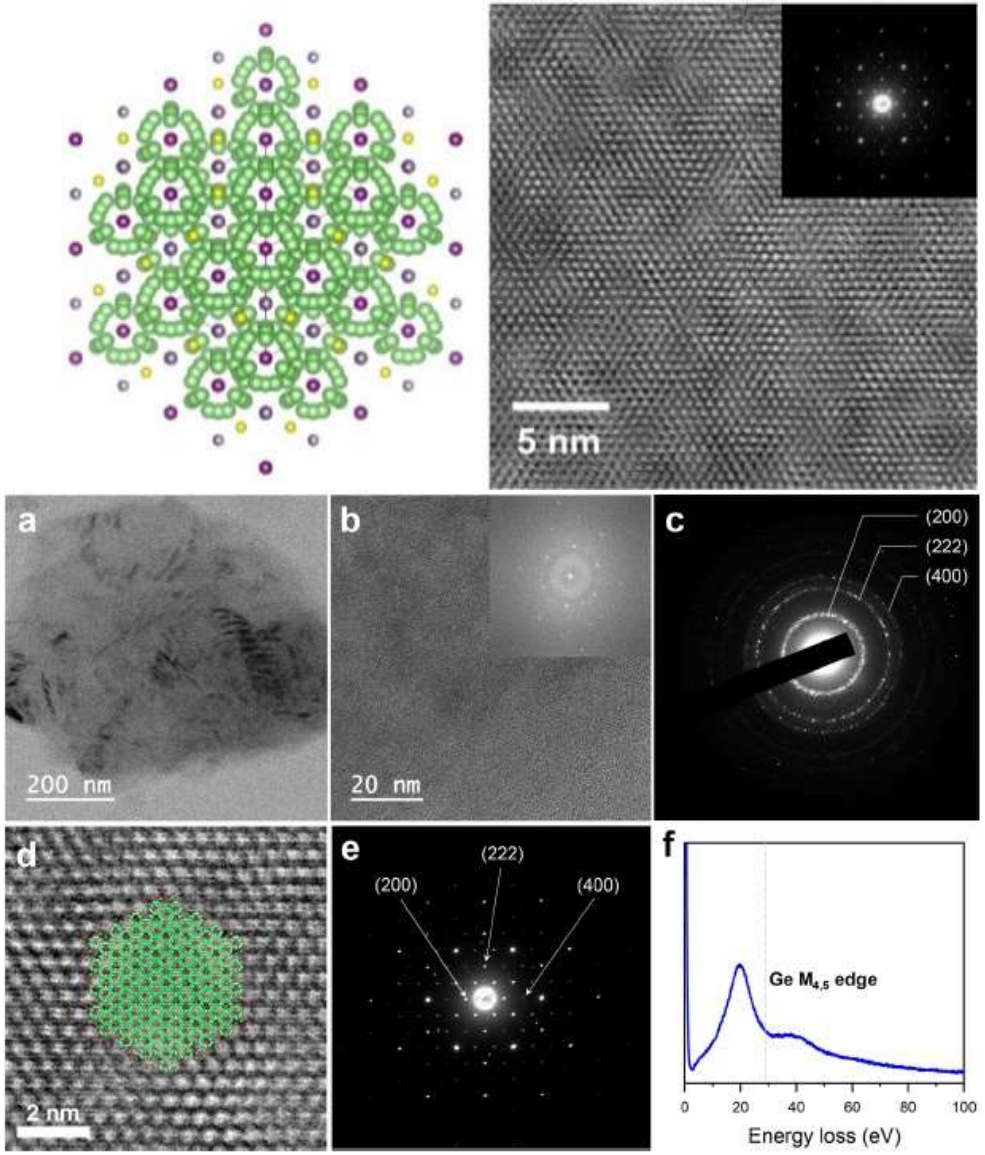
Yong Bae Song, Dong Hyeon Kim, Hiram Kwak, Daseul Han, Sujin Kang, Jong Hoon Lee, Seong-Min Bak, Kyung-Wan Nam, Hyun-Wook Lee,* and Yoon Seok Jung*

Cite This: https://dx.doi.org/10.1021/acs.nanolett.0c01028 — Read Online

ACCESS | Metrics & More | Article Recommendations | Supporting Information

ABSTRACT: Owing to their high Li^+ conductivities, mechanical sinterability, and solution processability, sulfide Li argyrodites have attracted much attention as enablers in the development of high-performance all-solid-state batteries with practicability. However, solution-processable Li argyrodites have been developed only for a composition of Li_6PS_5X (X = Cl, Br, I) with insufficiently high Li^+ conductivities (~10^{-4} S cm^{-1}). Herein, we report the highest Li^+ conductivity of 0.54 mS cm^{-1} at 30 °C ($Li_{6.5}P_{0.5}Ge_{0.5}S_5I$) for solution-processable iodine-based Li argyrodites. A comparative investigation of three iodine-based argyrodites of unsubstituted and Ge- and Sn-substituted solution-processed Li_6PS_5I with varied heat-treatment temperature elucidates the effect of microstructural evolution on Li^+ conductivity. Notably, local nanostructures consisting of argyrodite nanocrystallites in solution-processed $Li_{6.5}P_{0.5}Ge_{0.5}S_5I$ have been directly captured by cryogenic transmission electron microscopy, which is a first for sulfide solid electrolyte materials. Specifically, the promising electrochemical performances of all-solid-state batteries at 30 °C employing $LiCoO_2$ electrodes tailored by the infiltration of $Li_{6.5}P_{0.5}Ge_{0.5}S_5I$–ethanol solutions are successfully demonstrated.

KEYWORDS: *Solid-state batteries, solid electrolytes, cryo-TEM, sulfides, solution process*



Provision of data

NANO LETTERS (May 5, 2020)
ACS Publications
American Chemical Society

Mel-Build

Publication of recent papers-2

THE JOURNAL OF PHYSICAL CHEMISTRY LETTERS
A JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
pubs.acs.org/JPCL
Letter

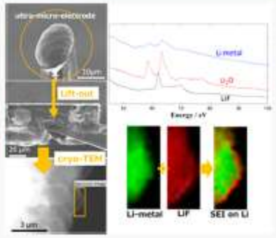
Characterization of Electrodeposited Li Metal by Cryo-Scanning Transmission Electron Microscopy/Electron Energy Loss Spectroscopy

Kei Nishikawa* and Keisuke Shinoda

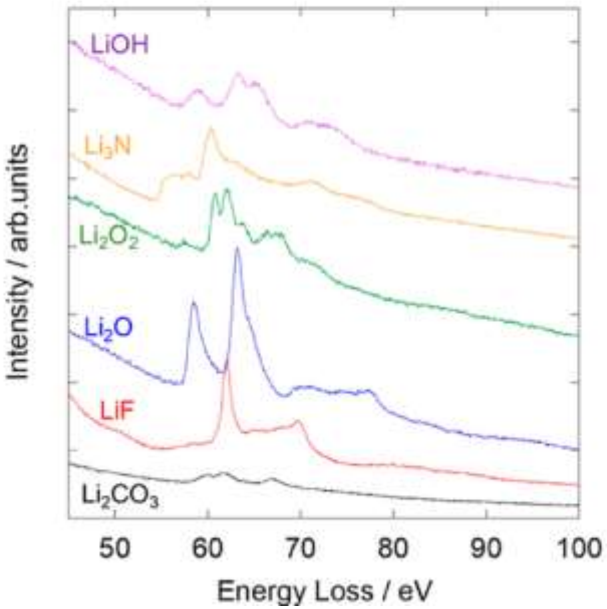
Cite This: J. Phys. Chem. Lett. 2021, 12, 3922−3927
Read Online

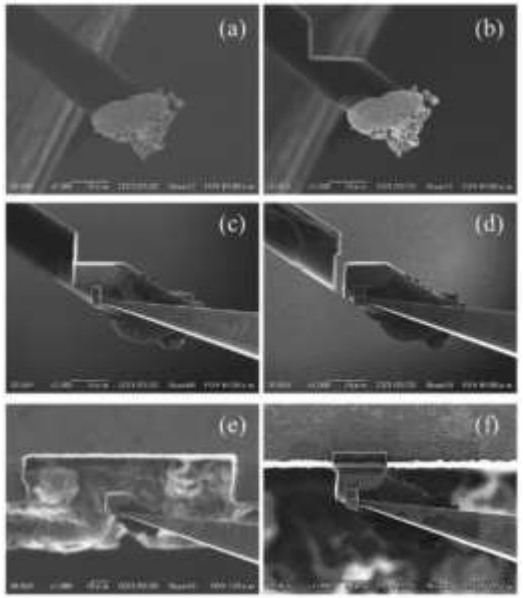
ACCESS | Metrics & More | Article Recommendations | Supporting Information

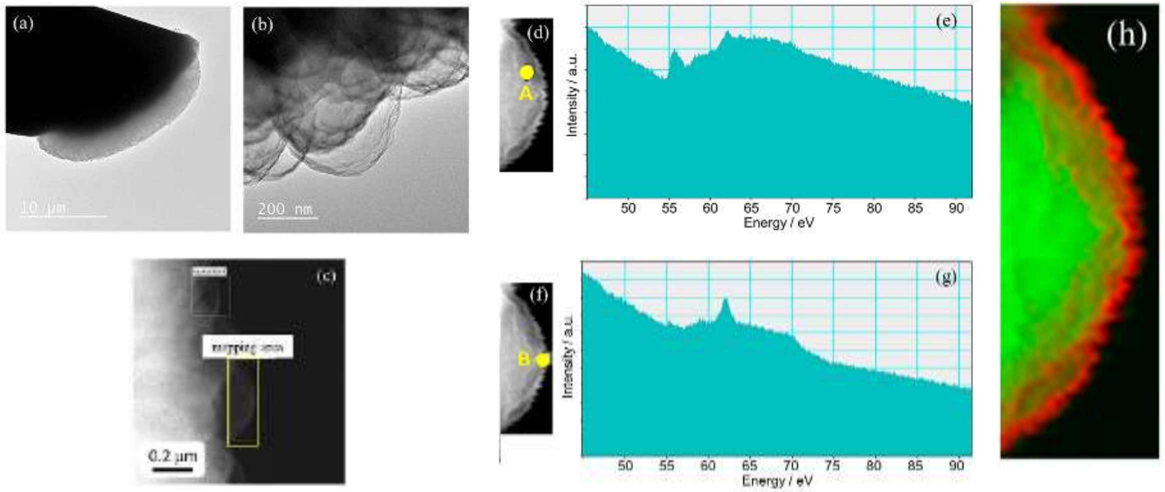
ABSTRACT: The Li metal anode is a promising key component for next-generation high-energy-density batteries. Understanding the charge/discharge mechanism of Li metal is therefore necessary for the effective utilization of Li metal anodes in commercial batteries. In this study, scanning transmission electron microscopy (STEM) combined with electron energy loss spectroscopy (EELS) was conducted to reveal the chemical state of the Li metal anode surface. Cryogenic techniques and ultramicroelectrodes (UMEs) enabled the observation of electrodeposited Li metal on the nanometer scale. The chemical compositions of several surface layers were revealed by cryo-STEM-EELS analysis, and these measurements gave crucial information regarding the surface layer of the electrodeposited Li metal.



Li metal is a particularly attractive material for use as the negative electrode of next-generation batteries because the theoretical gravimetric capacity of Li metal is significantly larger than that of the graphite anode currently employed in Li-ion batteries. As a result, many researchers have attempted the application of Li metal negative electrodes to Li−S batteries and all-solid-state Li batteries.[1−3] However, dendrite formation is a significant issue that inhibits the commercialization of Li metal batteries, as Li dendrites trigger the short-circuit phenomenon during the charging and discharging operations. It is therefore necessary to overcome the Li dendrite problem to develop large energy density batteries, which is crucial for establishing photoelectron spectroscopy (XPS).[11,17,19−21] XPS can detect the chemical bonding of surface chemical species; therefore, XPS is one of the standard analytical techniques employed in battery materials research. Furthermore, since Li is the lightest metal, detection of its chemical state is challenging; however, this can be achieved by XPS for the Li metal surface and for a number of other Li compounds. Due to the low spatial resolution, however, alternative techniques have been examined. For example, energy dispersive X-ray spectroscopy (EDS) is often combined with scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to obtain a signal from a smaller area than that required for XPS. It is also







Provision of data

The Journal of Physical Chemistry Letters
(April 16, 2021)
American Chemical Society

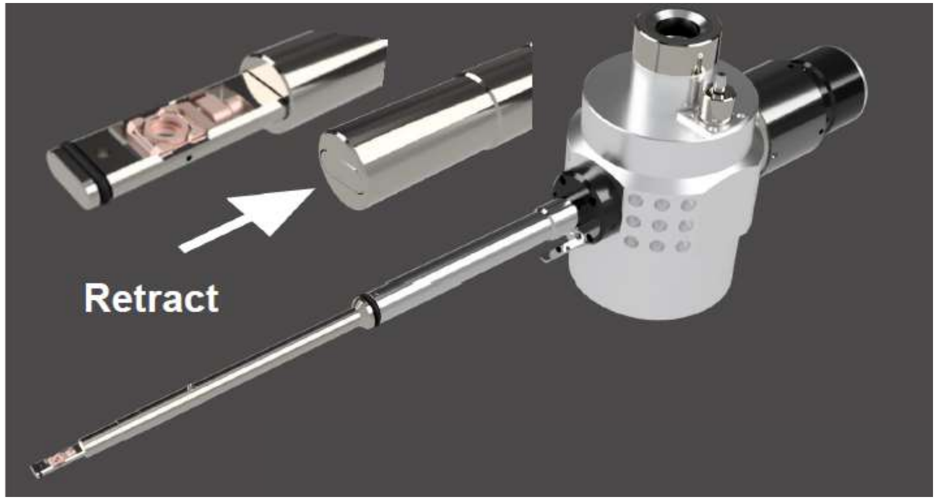
Developing Progress

Double tilt LN_2 Atoms Defend Holder for TFS

Under developing products

Product Summary

Holder



Basic Item(Recommended package) *Additional options are available

Temp. control unit

safety standard

＊Certification scheduled in 2020

Safety Operation Stand

Easy and safety sample mounting in glove box

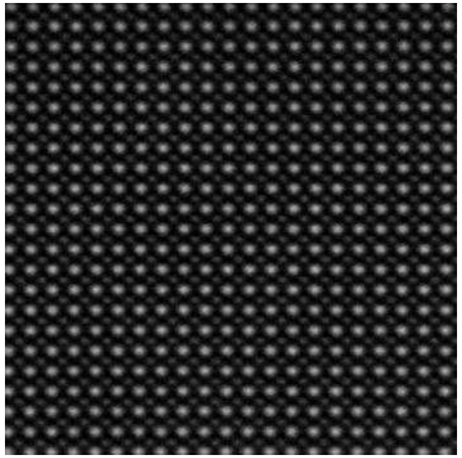
Torque Driver

Manages the torque of screw fixing.
Type:10mN

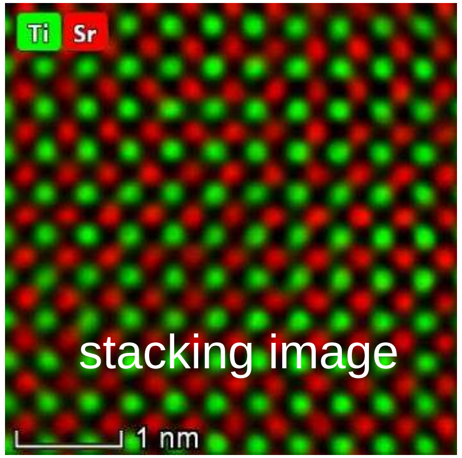
Specification

●**Mechanism**	:	**Double tilt / LN_2 Cooling / Vacuum Transfer**
●**Ultimate temp.**	:	Guarantee temp.-155℃(-160±5℃) ＊
●**Arrival time**	:	60 min
●**X tilt**	:	± 30 degree @ ST PP
●**Y tilt**	:	± 20 degree @ ST PP
●**Resolution**	:	0.24nm Au Nano Particle TEM FFT 002 observation (Factory check specification)
●**Transfer**	:	Ultimate vacuum inside the storage <1X10-3Pa
●**Drift Rate**	:	6 nm/min (to be improved)

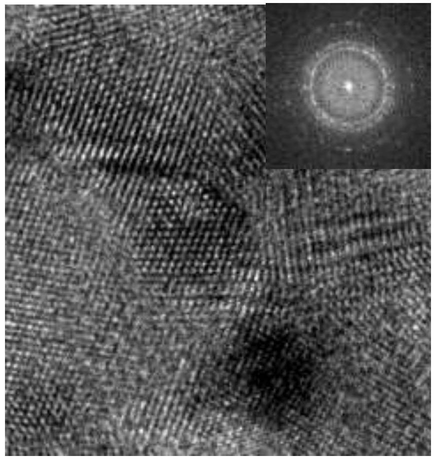
STO HR STEM by DCFI



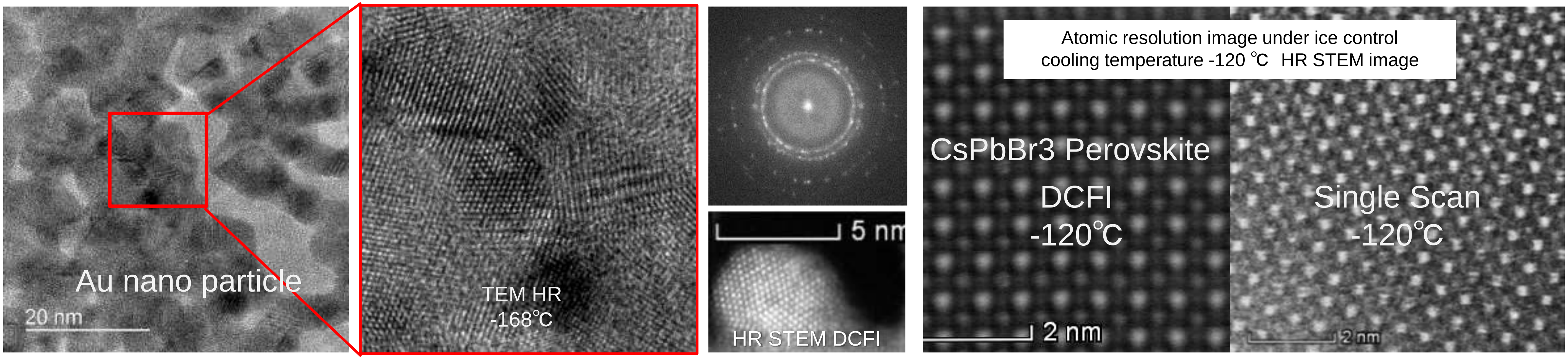
STO HR EDS mapping



Au nano Particle



Data summary HR imaging

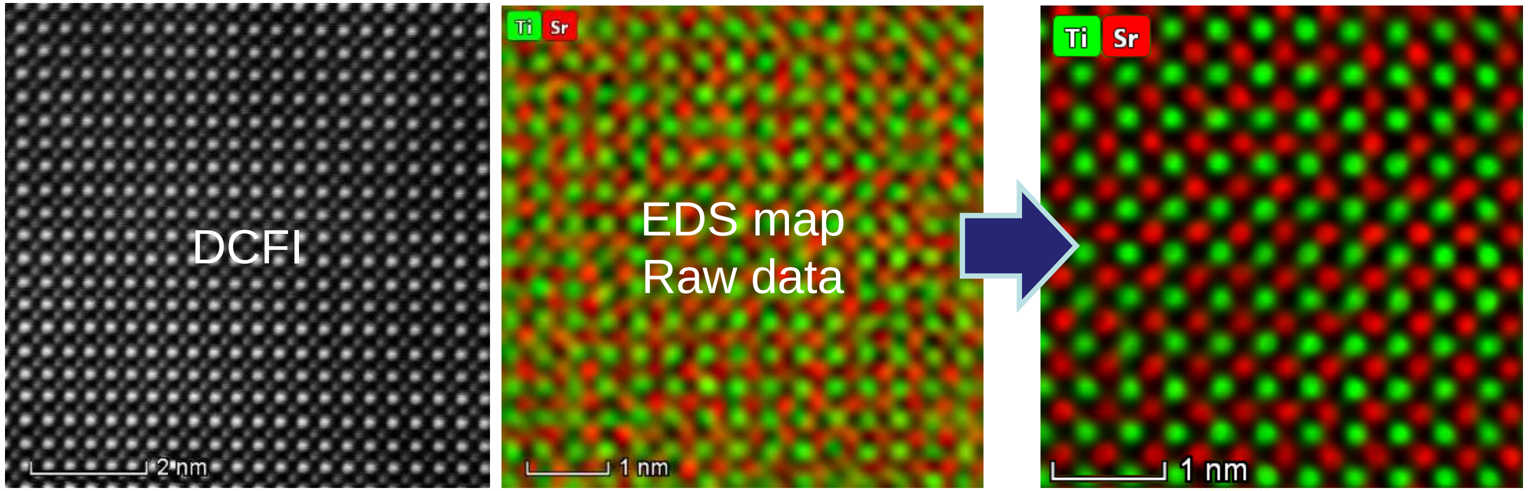


Test summary

•Sample	:	AU nano particle , CsPbBr3 and $SrTiO_3$
•Magnification	:	HR image TEM and STEM
•Equipment	:	Titian Cube in Kyushu University
•Start time	:	After reached -160 ℃
•Collaborators	:	Associate professor Hikaru Saito

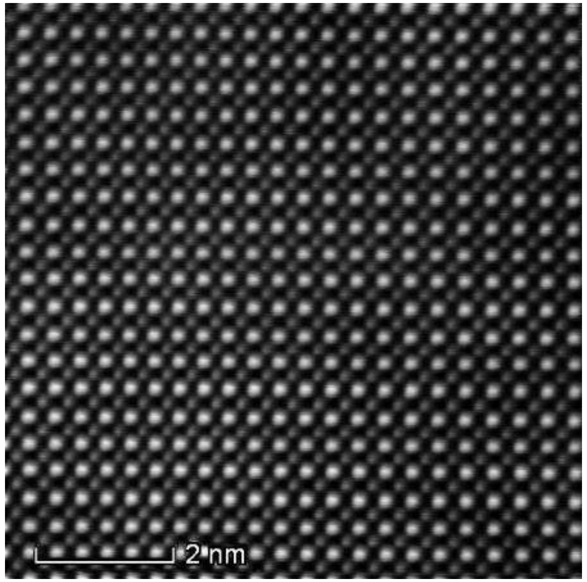
STO HR STEM image with DCFI and EDS mapping @ -160℃

After filtering and stacking image



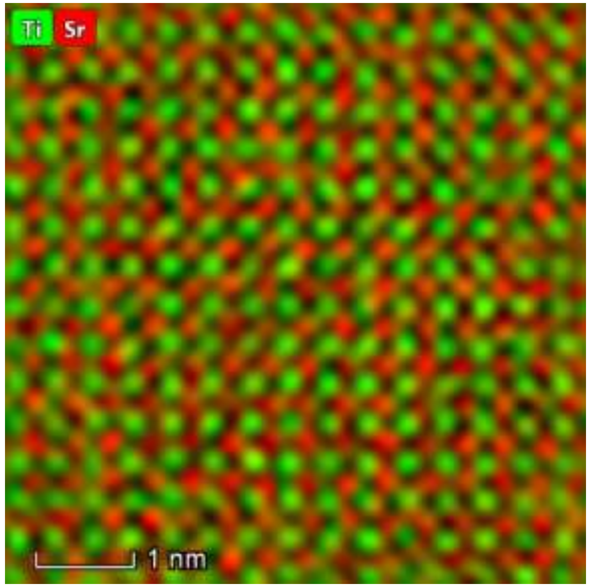
$SrTiO_3$ HR TEM image

STO HR STEM image with DCFI

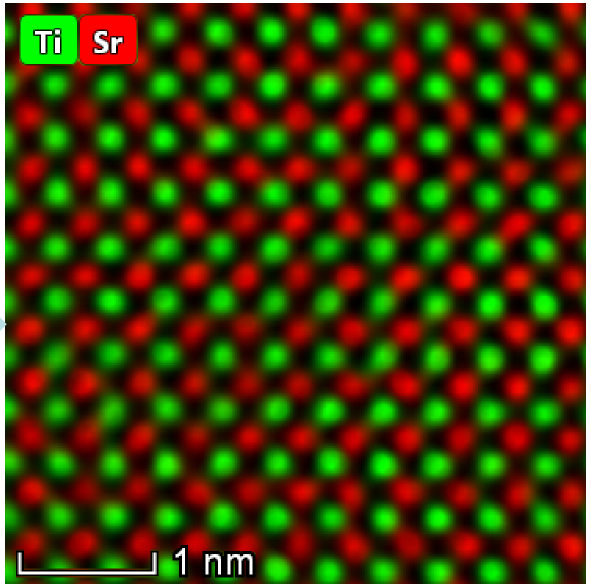


Video during EDS with drift correction by iPhone video

STO EDS mapping Raw data



After filtering and stacking image



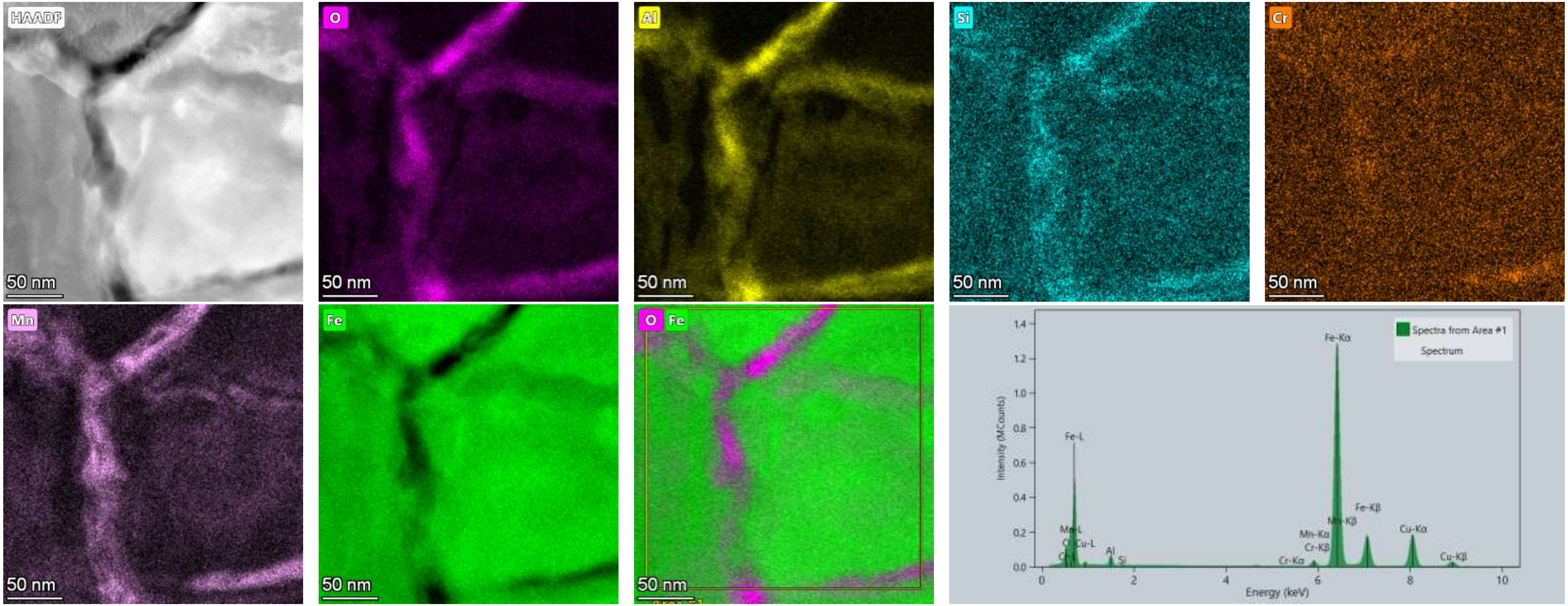
Note:
It has a good stable performance as a cooling holder.
We have succeeded in earning EDS mapping, However it is not easy.
It is difficult to get clean mapping data because the drift collection threshold is exceeded in 3 minutes.
I think there is a possibility that it can be solved by software.
The holder also continuous developing to improve stability.

Test summary

●Sample	:	$SrTiO_3$
●Magnification	:	HR STEM image 25M/50M
●Equipment	:	Titian Cube in Kyushu University
●Start time	:	After reached -160 ℃
●Collaborators	:	Associate professor Hikaru Saito

EDS mapping with Middle Resolution

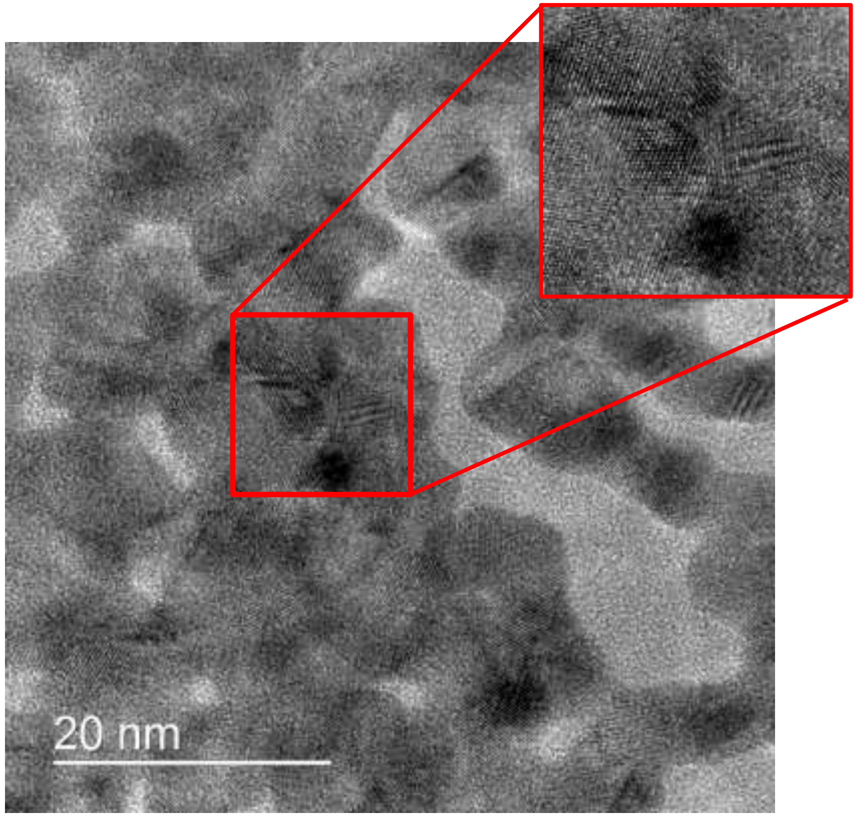
Sample : steal

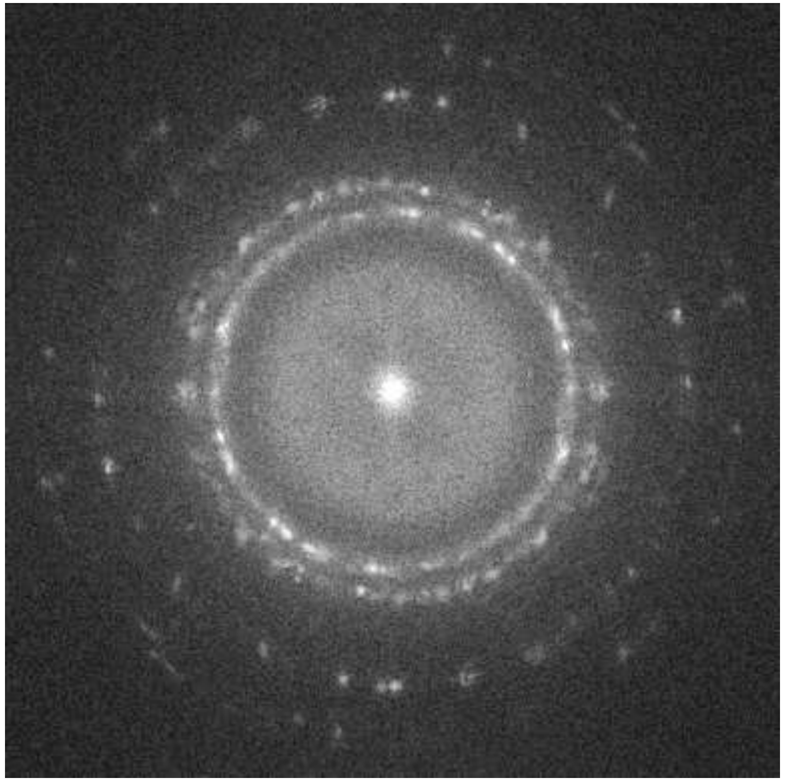


EDS	Image size	Acq time	Probe Current	Drift Correction	Display Type
	512 × 512	8min	3.5nA	On	Net Intensity(Filtered)

Au nano particle TEM HR image

Resolution specification detail

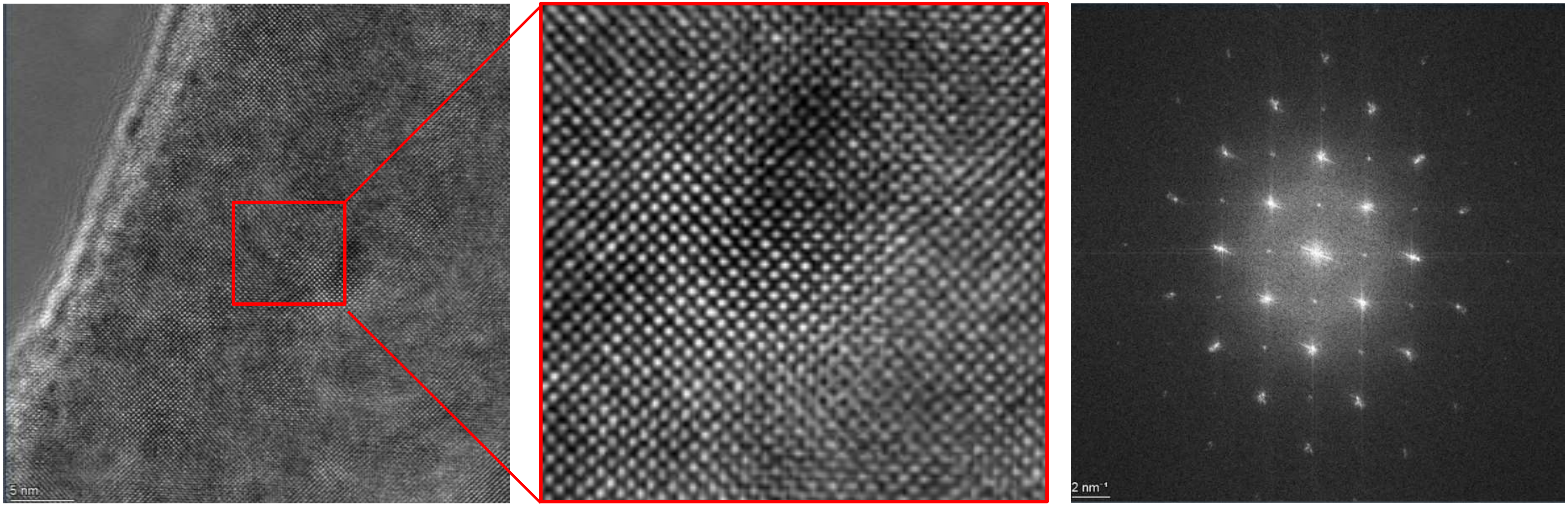




Resolution specification detail and note.

We will check the high resolution image of TITAN at Kyushu University
Normally cooling holder is very sensitive issue of environment.
so resolution image different from TEM room condition.

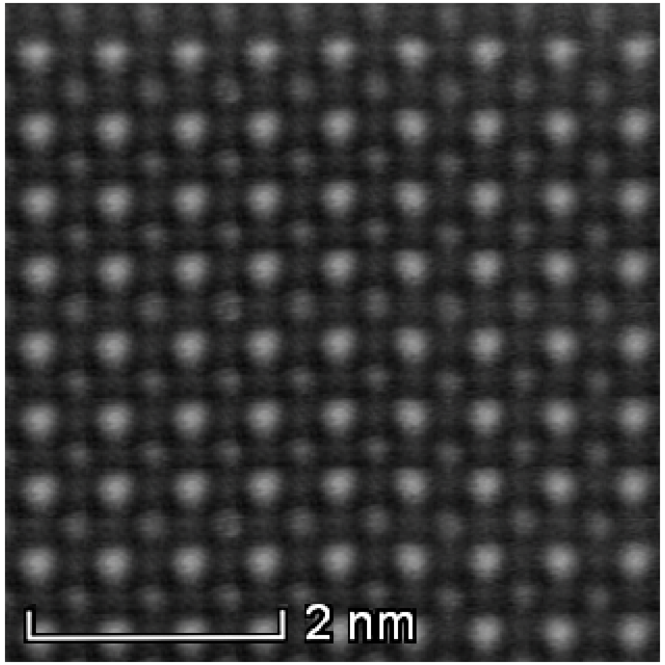
SrTiO3 HR TEM image

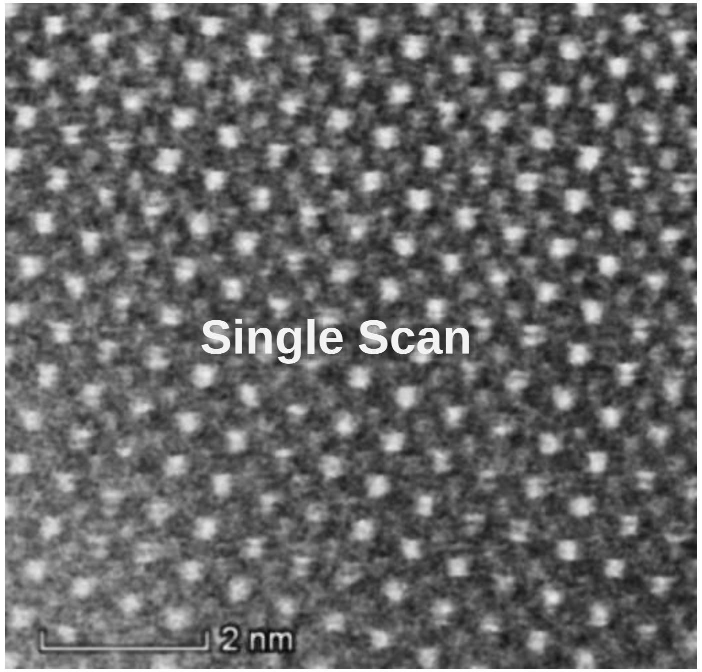


Test summary

●**Place**	:	Nanoport Japan(Shinagawa Ward,Tokyo)
●**Sample**	:	$SrTiO_3$
●**Equipment**	:	Talos F200X
●**Start time**	:	After reached -162 ℃

CsPbBr3 Perovskite HR STEM image





Test summary

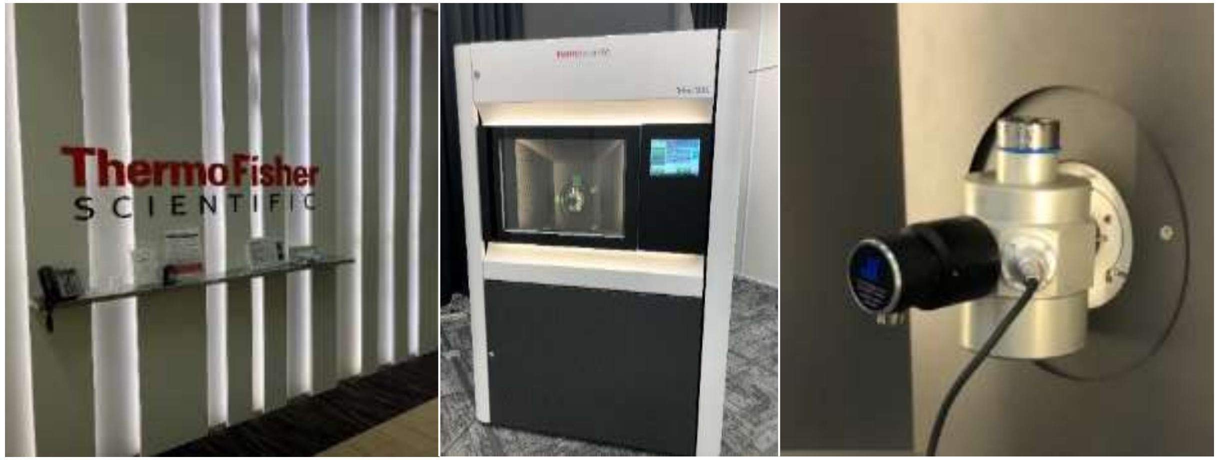
●**Sample**	:	CsPbBr3 Perovskite From the beginning, the temperature was controlled at -120 ℃ to suppress ice formation.
●**Equipment**	:	FEI titian overlay image by DCFI
●**Start time**	:	After reached -120 ℃
●**Tilt**	:	Y tilt : 1.3 degree X tilt : 3.3 degree

Development progress Summary-1

Development Topics

For Material Science TEM System / Workflow

Talos F200X + Double tilt LN_2 Atoms Defend Holder



Test summary

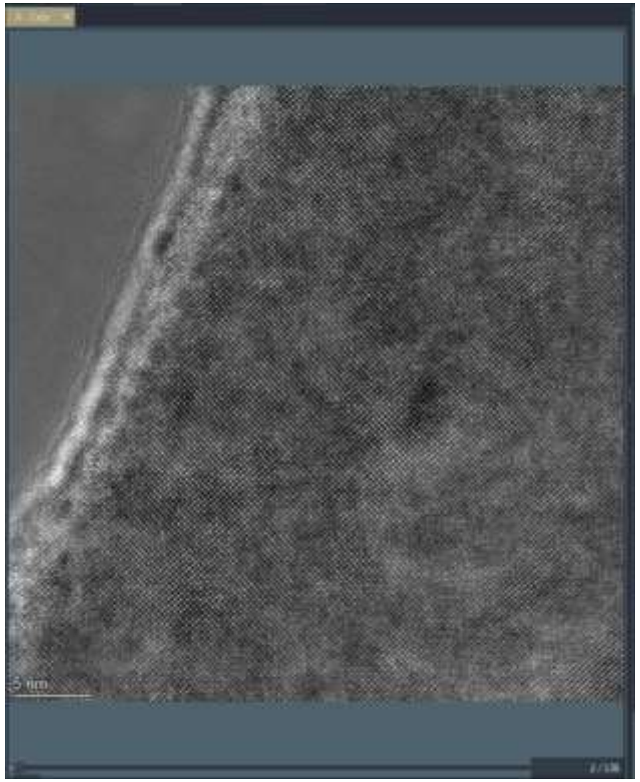
●**Date**	:	June 4th-5th, 2020
●**Place**	:	Nanoport Japan(Shinagawa Ward,Tokyo)
●**Sample**	:	$SrTiO_3$
●**Equipment**	:	Talos F200X
●**Start time**	:	After reached -162 ℃

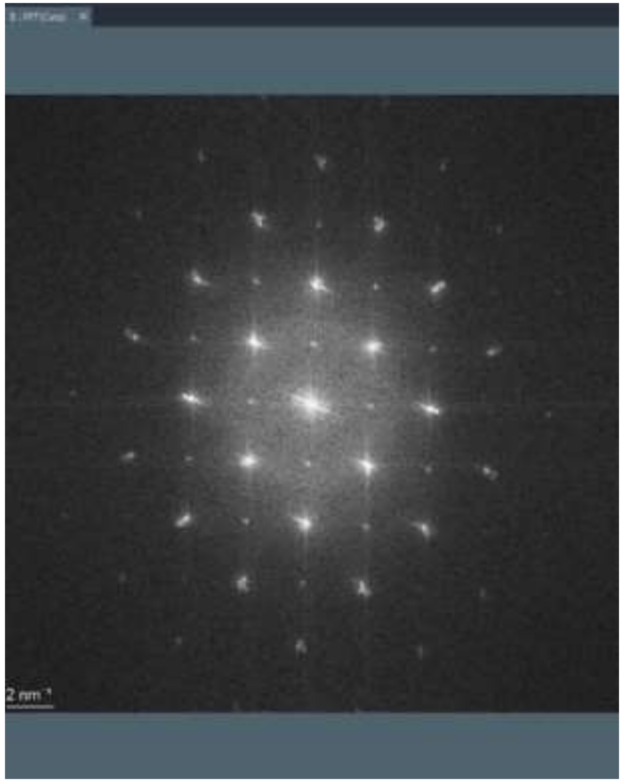
Development progress Summary-2

TEM

Test summary

●**Series acquisition**	:	1024×1024 200ms 136 frames without use of piezo stage
●**Video output**	:	1024×1024 5 fps



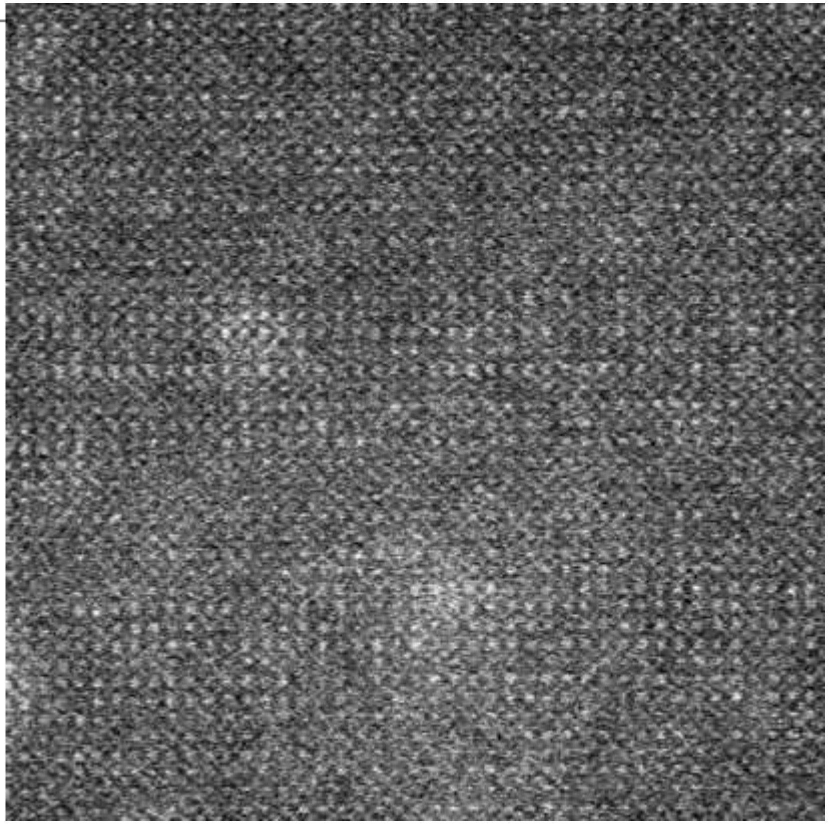


Development progress Summary-3

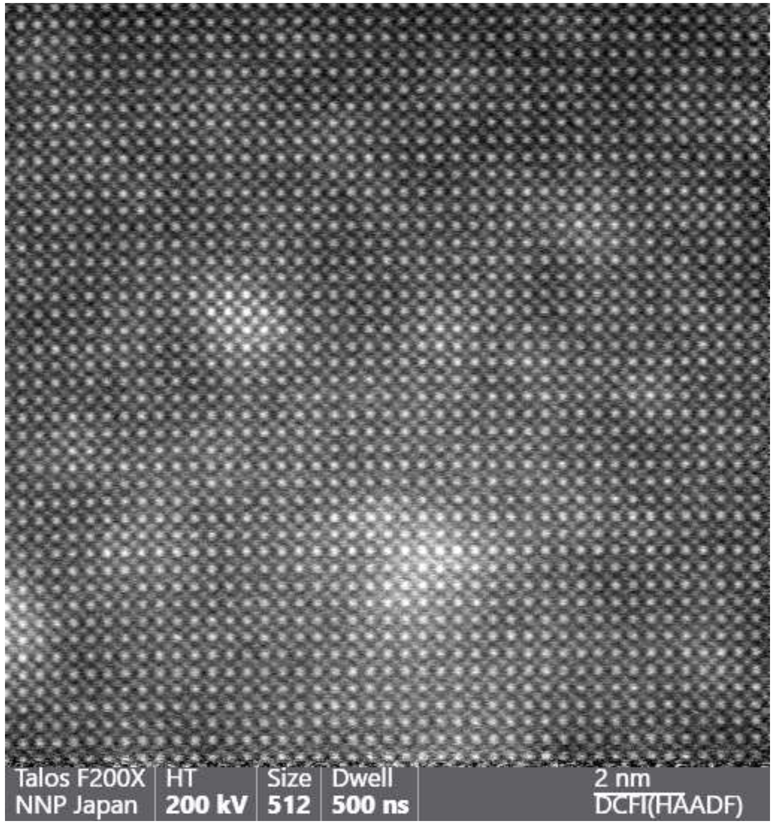
STEM

Test summary

●**Series acquisition**	:	512×512 500ns 30 frames Without use of Piezo stage
●**Video output**	:	512×512 1fps



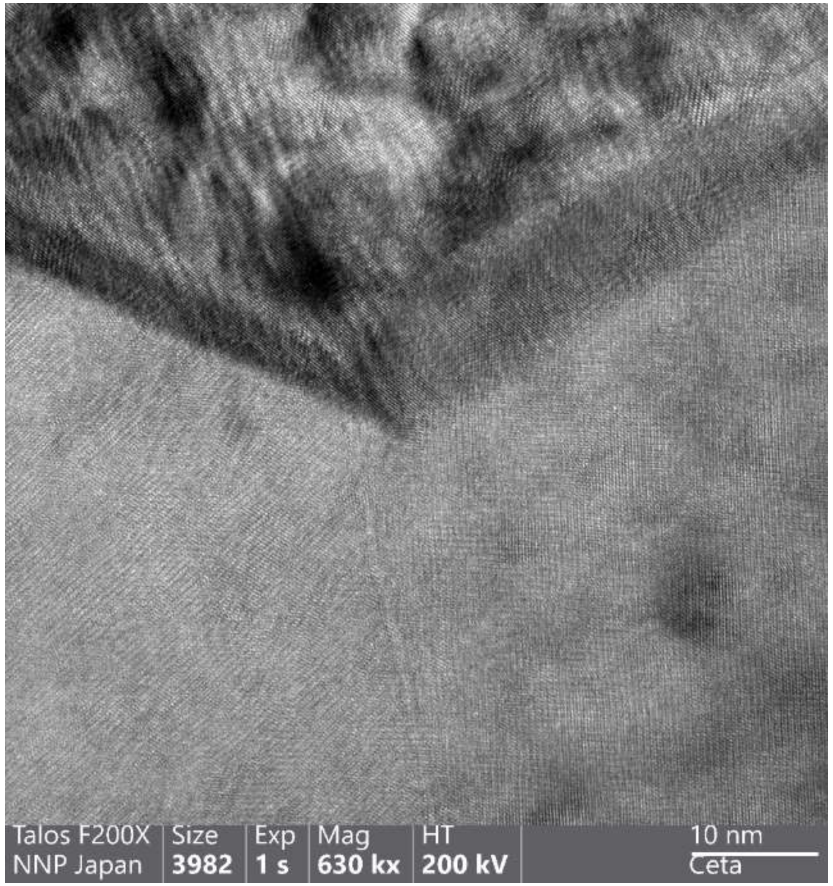
DCFI30



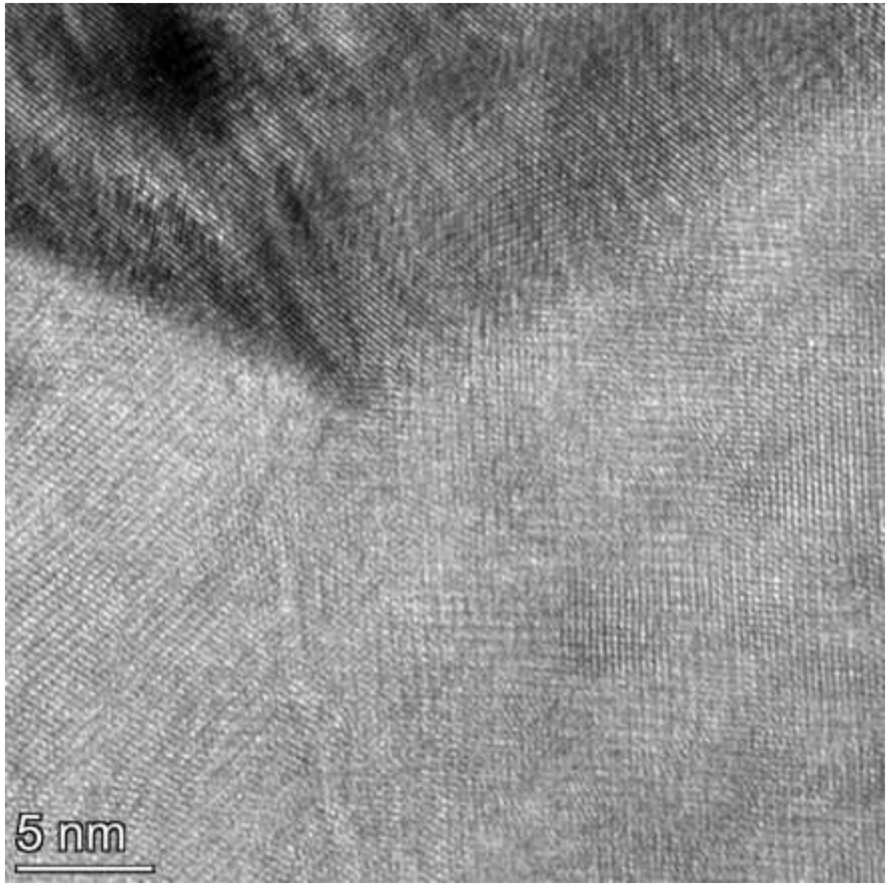
Development progress Summary-4

TEM Single acquisition

Without use of Piezo stage

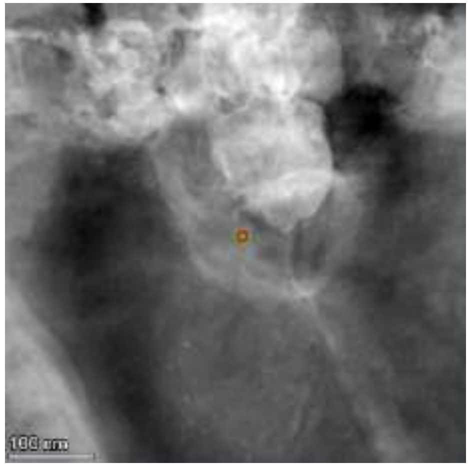


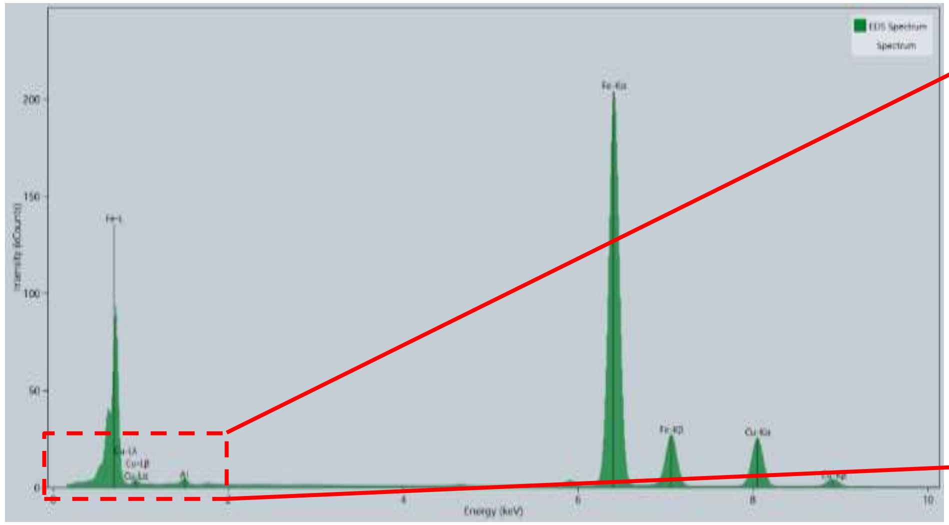
Digital Zoom



Development progress Summary-5

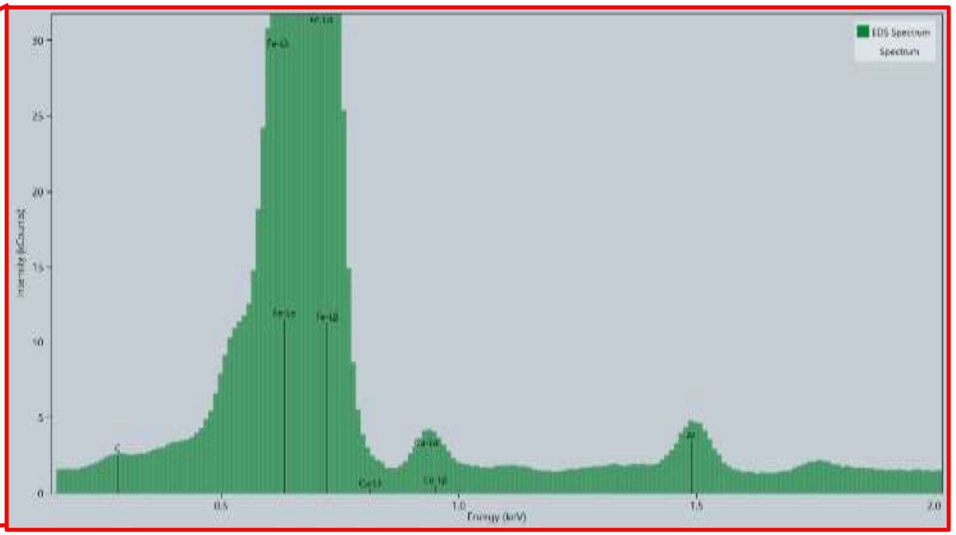
EDS point analysis





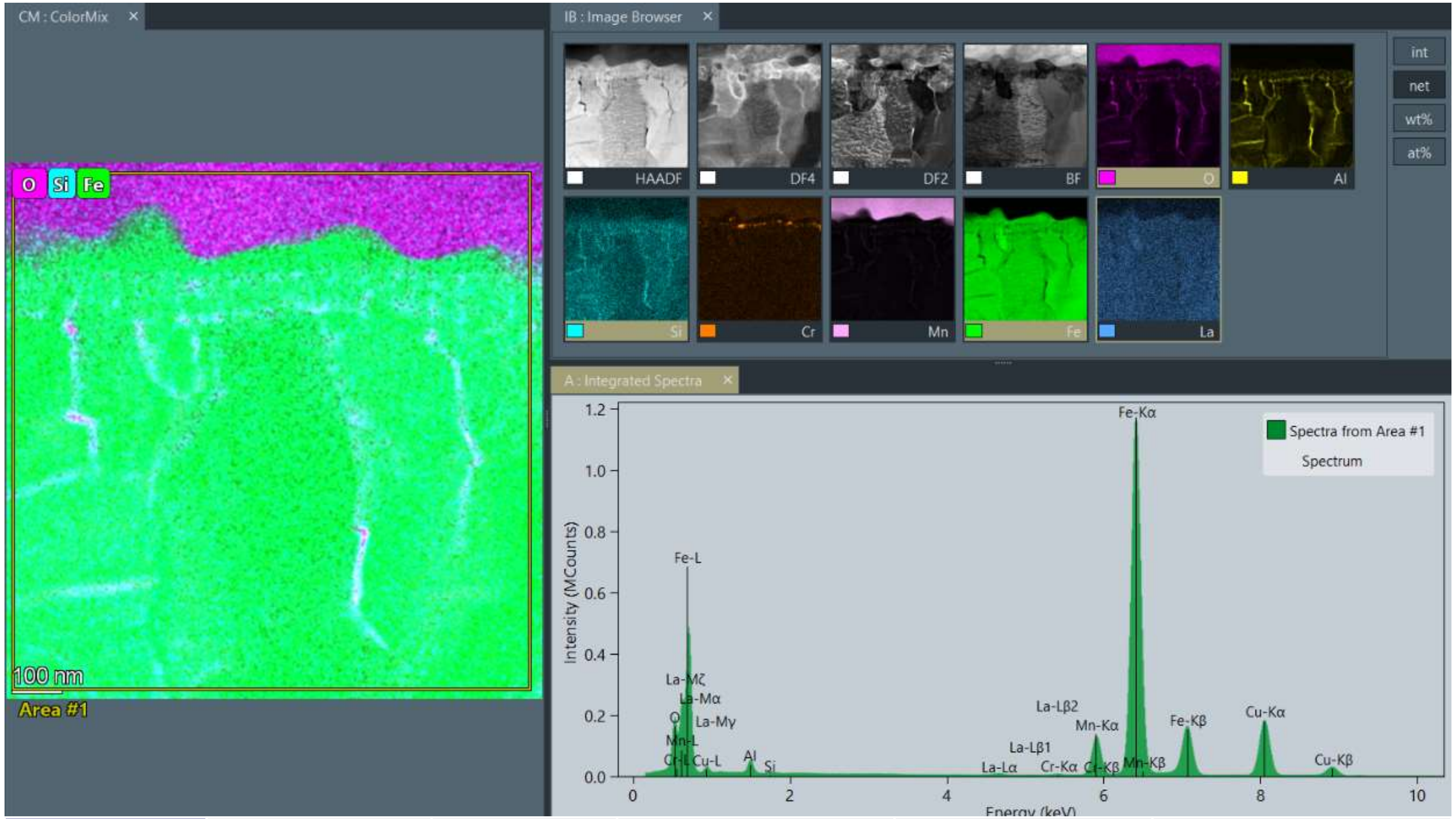
Clip: BeCu

Beam current 1nA
Acquisition time 2min
No contamination. Cu comes from BeCu of clip.



Development progress Summary-6

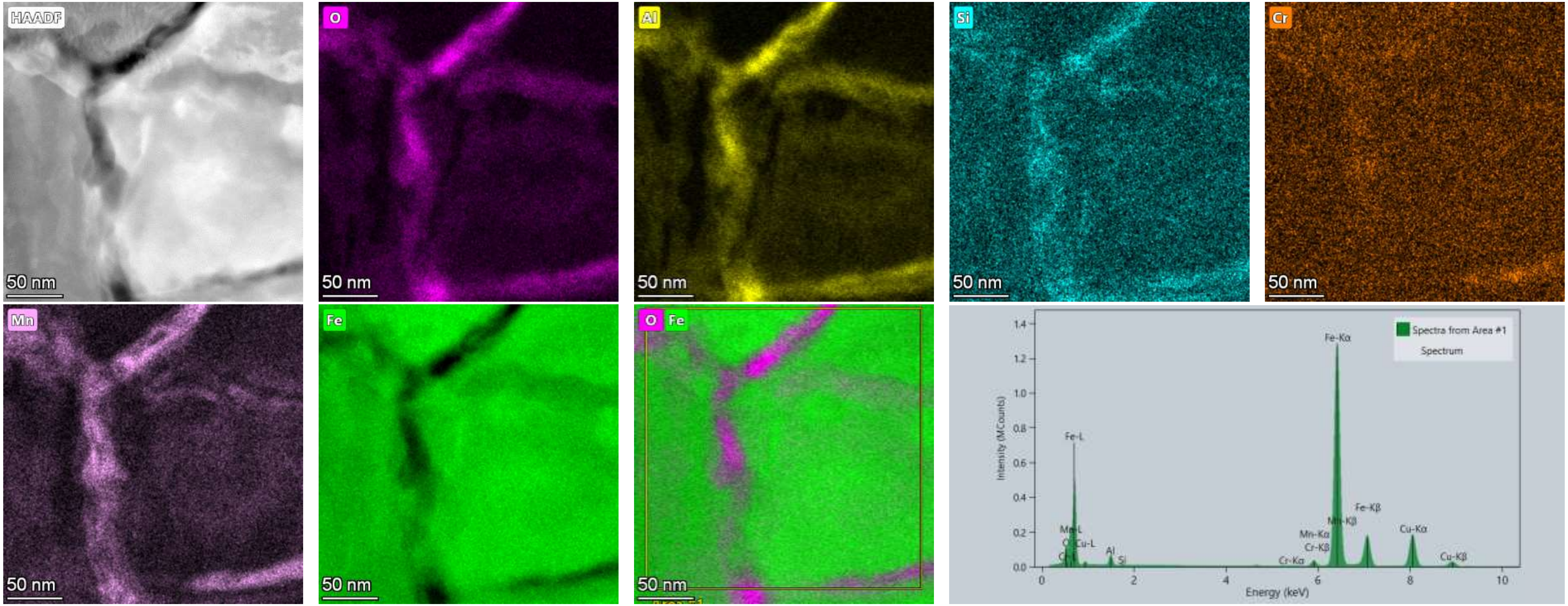
EDS mapping



EDS	Image size	Acq time	Probe Current	Drift Correction	Display Type
	512 × 512	8min	3.5nA	On	Net Intensity(Filtered)

Development progress Summary-7

EDS mapping



EDS	Image size	Acq time	Probe Current	Drift Correction	Display Type
	512 × 512	8min	3.5nA	On	Net Intensity(Filtered)

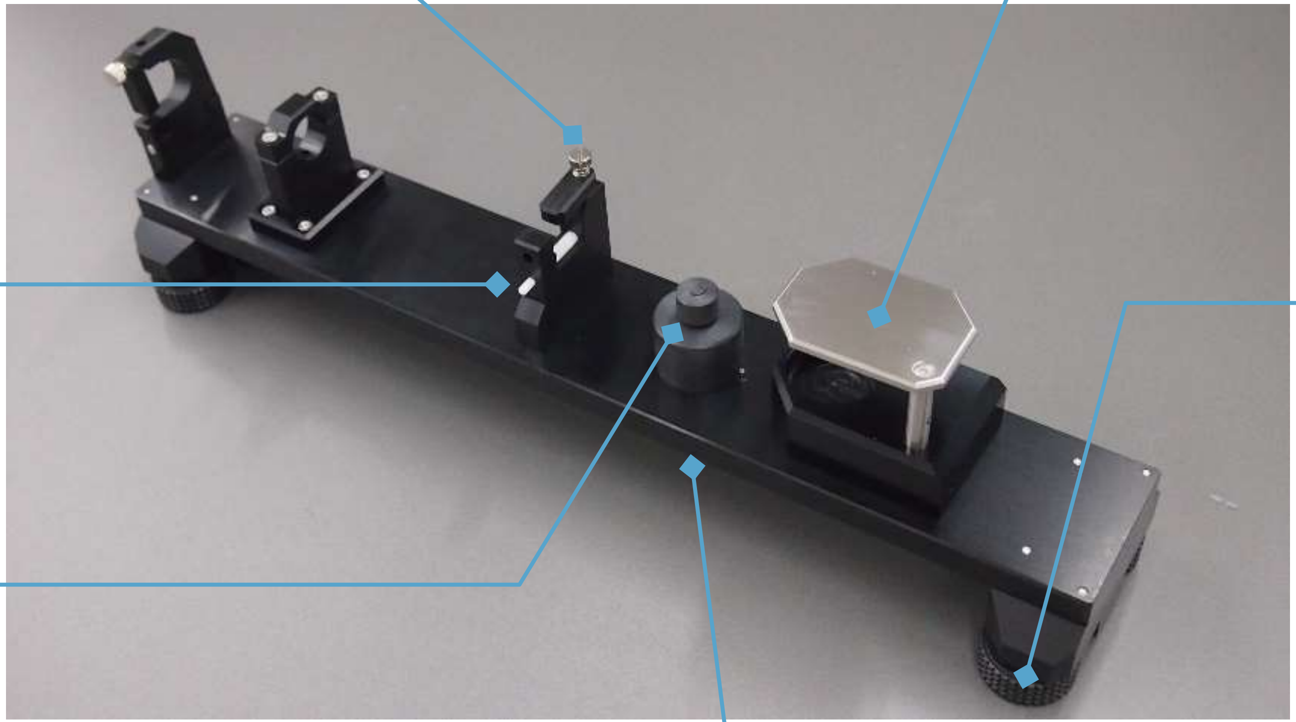
Safety Operation Stand

Working inside the glove box is very difficult,
A stable workbench is required when setting the sample

Holder holding plate:
Since you firmly clamp the holder, you can work safely until the work is completed.

Release button:
You can easily remove the holder from the pedestal with the release button

Mini jack:
We hold the specimen pedestal from below, weak strength, so you can fix the specimen safely. (Fitting sample holder ring from above, necessary for this proposal)

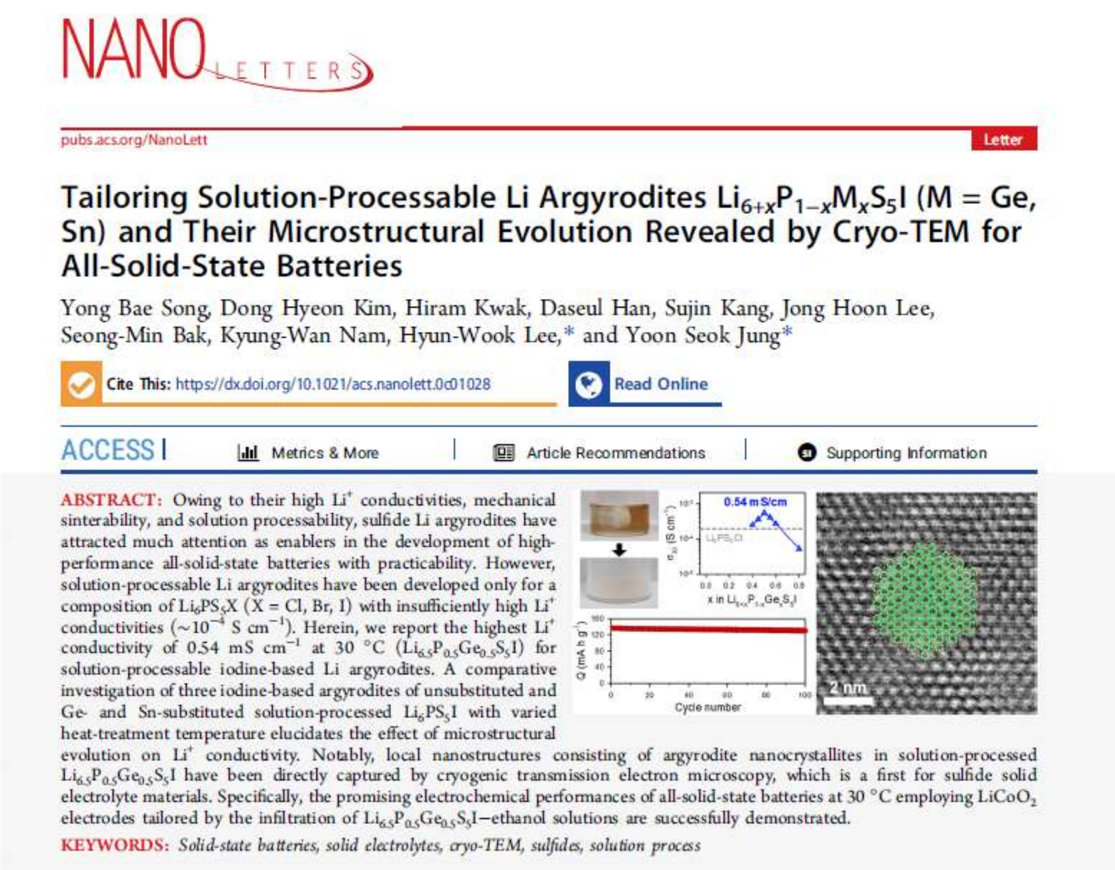


Palm rest:
You can rotate 360°, you can rotate it to your favorite position, when handling the sample when installing the sample.
Since the hand is fixed, you can attach the sample in a stable condition.

Height adjustment leg:
Since the height can be independently adjusted in 4 places, it can be adjusted so that stable work can be performed even with a slight distortion and inclination.

Bridge structure:
Since it has a bridge structure, it can work directly under an optical microscope.
(Some lights can not be used)

Publication of recent papers-1



NANO LETTERS

pubs.acs.org/NanoLett Letter

Tailoring Solution-Processable Li Argyrodites $Li_{6+x}P_{1-x}M_xS_5I$ (M = Ge, Sn) and Their Microstructural Evolution Revealed by Cryo-TEM for All-Solid-State Batteries

Yong Bae Song, Dong Hyeon Kim, Hiram Kwak, Daseul Han, Sujin Kang, Jong Hoon Lee, Seong-Min Bak, Kyung-Wan Nam, Hyun-Wook Lee,* and Yoon Seok Jung*

Cite This: https://dx.doi.org/10.1021/acs.nanolett.0c01028 Read Online

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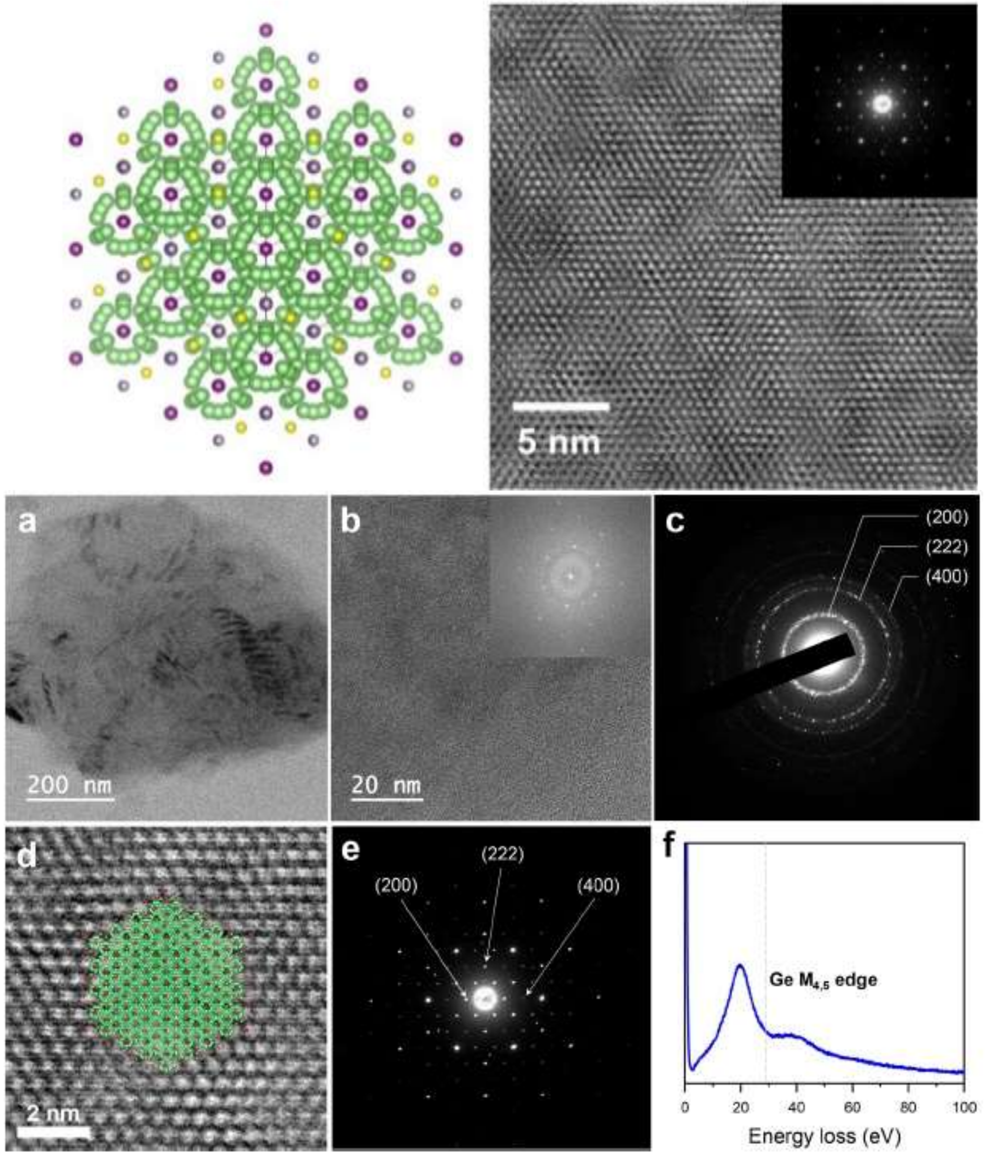
ABSTRACT: Owing to their high Li^+ conductivities, mechanical sinterability, and solution processability, sulfide Li argyrodites have attracted much attention as enablers in the development of high-performance all-solid-state batteries with practicability. However, solution-processable Li argyrodites have been developed only for a composition of Li_6PS_5X (X = Cl, Br, I) with insufficiently high Li^+ conductivities (~10^{-4} S cm^{-1}). Herein, we report the highest Li^+ conductivity of 0.54 mS cm^{-1} at 30 °C ($Li_{6.5}P_{0.5}Ge_{0.5}S_5I$) for solution-processable iodine-based Li argyrodites. A comparative investigation of three iodine-based argyrodites of unsubstituted and Ge- and Sn-substituted solution-processed Li_6PS_5I with varied heat-treatment temperature elucidates the effect of microstructural evolution on Li^+ conductivity. Notably, local nanostructures consisting of argyrodite nanocrystallites in solution-processed $Li_{6.5}P_{0.5}Ge_{0.5}S_5I$ have been directly captured by cryogenic transmission electron microscopy, which is a first for sulfide solid electrolyte materials. Specifically, the promising electrochemical performances of all-solid-state batteries at 30 °C employing $LiCoO_2$ electrodes tailored by the infiltration of $Li_{6.5}P_{0.5}Ge_{0.5}S_5I$–ethanol solutions are successfully demonstrated.

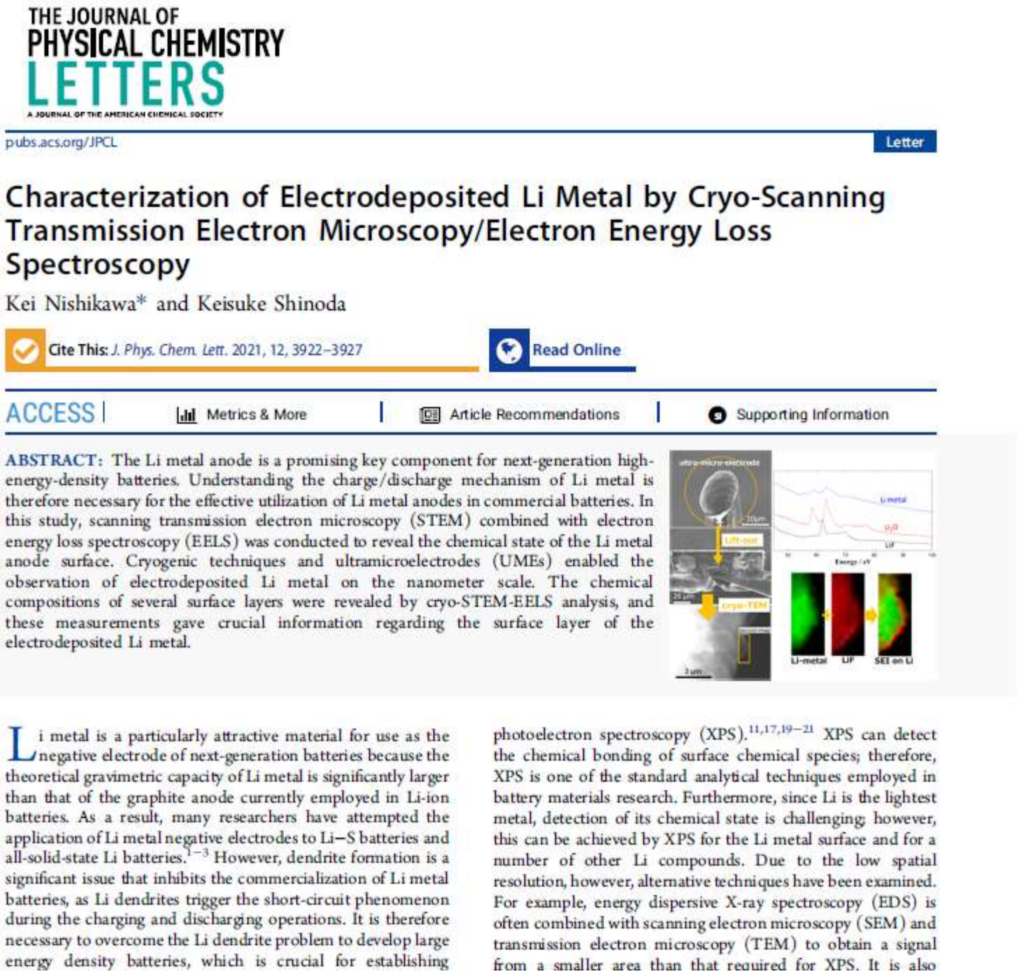
KEYWORDS: *Solid-state batteries, solid electrolytes, cryo-TEM, sulfides, solution process*



Provision of data

NANO LETTERS (May 5, 2020)
ACS Publications
American Chemical Society

Publication of recent papers-2


THE JOURNAL OF PHYSICAL CHEMISTRY LETTERS
A JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

pubs.acs.org/JPCL — Letter

Characterization of Electrodeposited Li Metal by Cryo-Scanning Transmission Electron Microscopy/Electron Energy Loss Spectroscopy

Kei Nishikawa* and Keisuke Shinoda

Cite This: *J. Phys. Chem. Lett.* 2021, 12, 3922−3927 — Read Online

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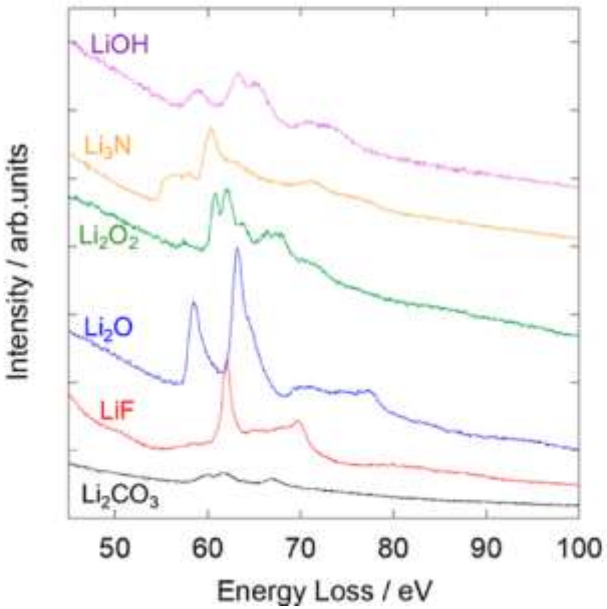
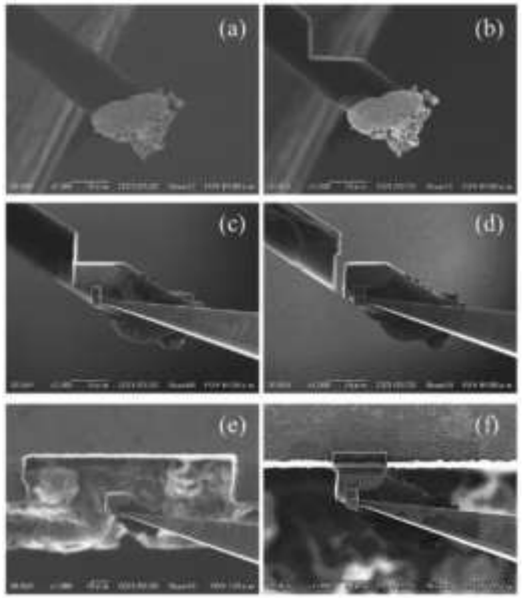
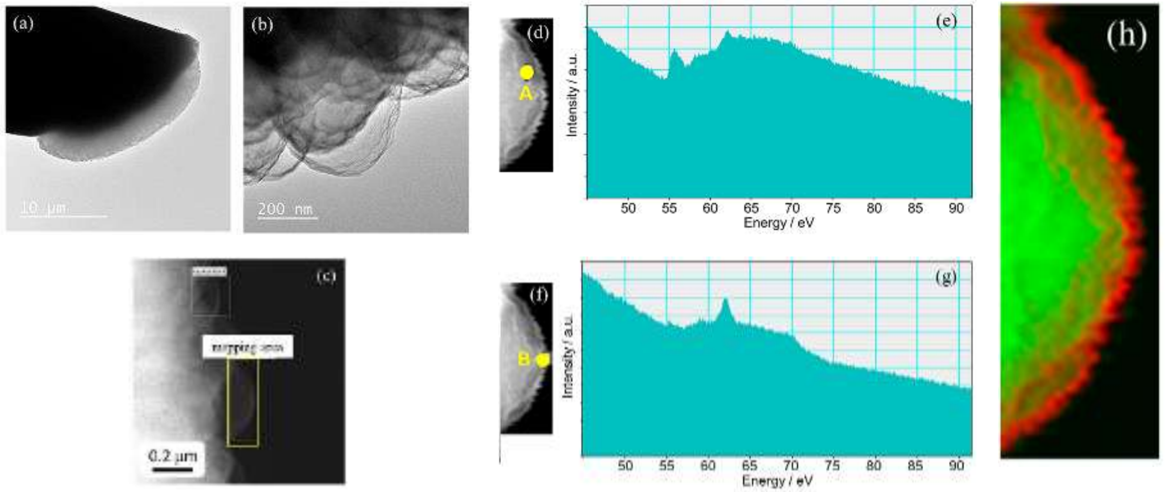
ABSTRACT: The Li metal anode is a promising key component for next-generation high-energy-density batteries. Understanding the charge/discharge mechanism of Li metal is therefore necessary for the effective utilization of Li metal anodes in commercial batteries. In this study, scanning transmission electron microscopy (STEM) combined with electron energy loss spectroscopy (EELS) was conducted to reveal the chemical state of the Li metal anode surface. Cryogenic techniques and ultramicroelectrodes (UMEs) enabled the observation of electrodeposited Li metal on the nanometer scale. The chemical compositions of several surface layers were revealed by cryo-STEM-EELS analysis, and these measurements gave crucial information regarding the surface layer of the electrodeposited Li metal.

Li metal is a particularly attractive material for use as the negative electrode of next-generation batteries because the theoretical gravimetric capacity of Li metal is significantly larger than that of the graphite anode currently employed in Li-ion batteries. As a result, many researchers have attempted the application of Li metal negative electrodes to Li−S batteries and all-solid-state Li batteries.[1−3] However, dendrite formation is a significant issue that inhibits the commercialization of Li metal batteries, as Li dendrites trigger the short-circuit phenomenon during the charging and discharging operations. It is therefore necessary to overcome the Li dendrite problem to develop large energy density batteries, which is crucial for establishing photoelectron spectroscopy (XPS).[11,17,19−21] XPS can detect the chemical bonding of surface chemical species; therefore, XPS is one of the standard analytical techniques employed in battery materials research. Furthermore, since Li is the lightest metal, detection of its chemical state is challenging; however, this can be achieved by XPS for the Li metal surface and for a number of other Li compounds. Due to the low spatial resolution, however, alternative techniques have been examined. For example, energy dispersive X-ray spectroscopy (EDS) is often combined with scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to obtain a signal from a smaller area than that required for XPS. It is also







Provision of data

The Journal of Physical Chemistry Letters
(April 16, 2021)
American Chemical Society