

# **Non-destructive analysis results of the second fuel debris sample (Follow-up report) and fractionation results**

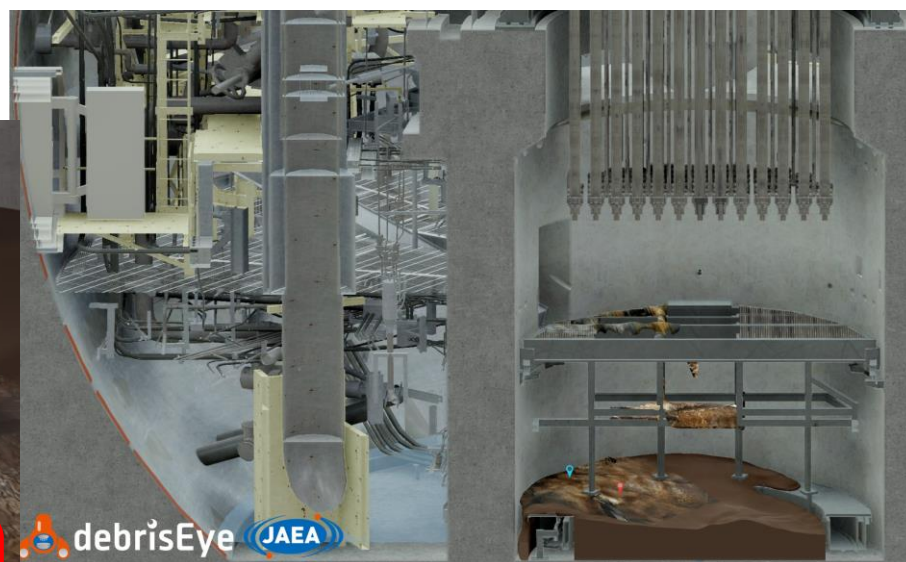
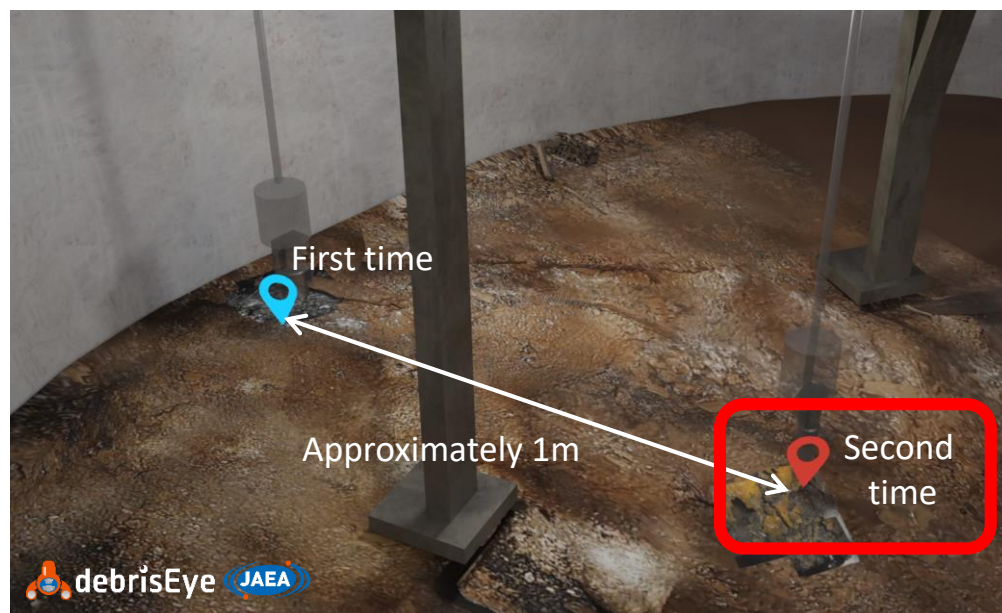
**August 28, 2025**

**Japan Atomic Energy Agency**

**Tokyo Electric Power Company Holdings, Inc.**

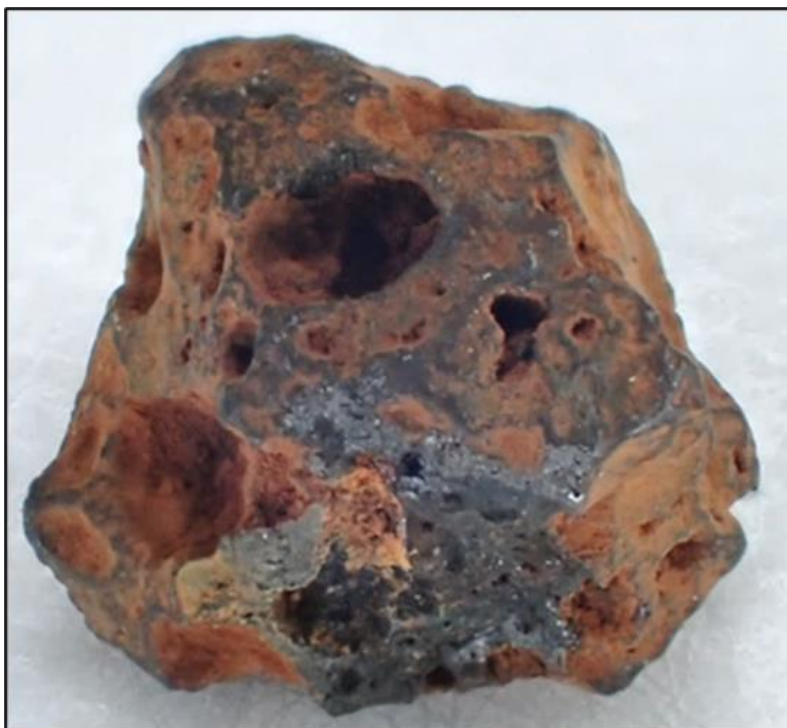
This report is related to the FY2025 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Characterization of Fuel Debris).

- On April 25, 2025, the second fuel debris sample taken during trial fuel debris retrieval was received at the JAEA Oarai Nuclear Engineering Institute's Irradiated Fuel Monitoring Facility (FMF).
- Out of all the non-destructive analyses, the results of external observations and  $\gamma$ -ray spectrometry, etc. were reported on at the Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting/Secretariat Meeting held on May 29, 2025. (Refer to the reference documents).
- At this meeting, a report will be given on detailed non-destructive analysis results (X-ray CT and SEM-WDX) and fractionation results.



The fuel debris sampling locations on the floor inside the Unit 2 pedestal

- The surface of the received sample is heterogeneous, chestnut-brown overall (a lighter color than the first sample) with black regions and pores on the surface.
- The size is approx. 5mm × approx. 4mm, and weight is 0.187g (including smaller fragments). Dose rate (γ-rays) is approx. 0.3mSv/h.
- γ-ray spectrometry detected AM-241 indicating the presence of fuel elements.



**(Front side:** Photographed from a 45° angle)



**(Rear side:** Photographed from a 45° angle)

Enlarged photo of external appearance of fuel debris sample

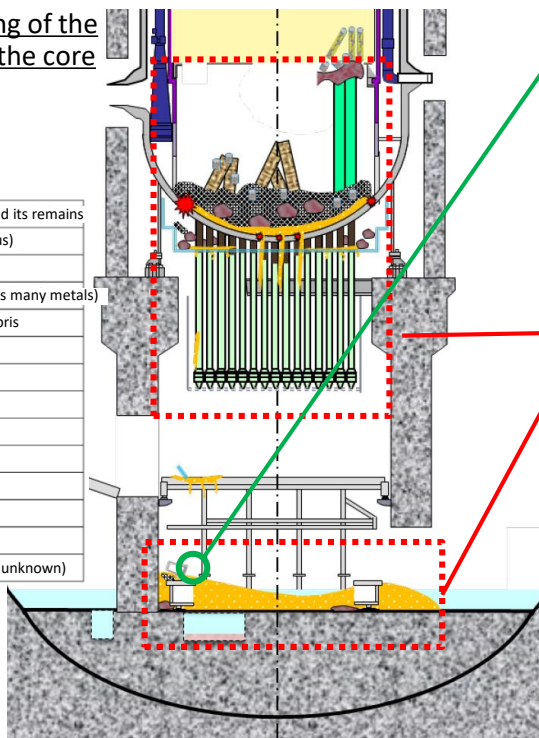
[1] JAEA, Fuel debris sample non-destructive analysis results, the Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting/Secretariat Meeting (138<sup>th</sup> meeting) held on May 29, 2025.

- By analyzing the obtained sample, grasp the condition of the sampled area to estimate the formation process of the fuel debris.
- ⇒ More precise estimation of the condition inside the core will become the basis for review of full-scale fuel debris retrieval to safely retrieve fuel debris and realize thoroughly managed stable storage.
- <Example of incorporating “estimation of the condition inside the core” into “review of fuel debris retrieval methods”>
  - Estimate hardness of fuel debris → select retrieval methods and tools
  - Possibility of criticality of fuel debris → review safety measures and storage methods

Estimated drawing of the condition inside the core

Unit 2<sup>[2]</sup>

	Residual fuel rod and its remains
	Oxide debris (porous)
	Particulate debris
	Fuel debris (contains many metals)
	Concrete mixed debris
	CRGT
	Damaged CRGT
	CRD
	CRD (debris inside)
	Shroud
	Pellets
	RPV damaged port
	Upper tie plate
	Deposits (materials unknown)



## 1. Grasping the condition of the sampled area

- Acquisition of **information tailored to decommissioning needs**
  - ✓ Grasp the type and concentration of major components (nuclide/element) in the sample and review the origin of each component
  - ✓ Grasp the content and distribution of fuel components in the sample

## 2. Estimation of formation process of fuel debris

- Estimation of fuel debris properties through **review of in-core environment during the accident**
  - ✓ Estimate the formation conditions of the sample based on microstructure, composition of constituent phases and crystal structure of phases including U in the sample.
  - ✓ Evaluate the surrounding of the sampled area based on the comparison of existing accident scenarios with the internal investigation results (evaluate based on the results of multiple future sample analyses)

[1] JAEA, Fuel debris sample non-destructive analysis results, the Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting/Secretariat Meeting (138<sup>th</sup> meeting) held on May 29, 2025.

[2] JAEA, FY2022 Report of the FY2022 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Grasping Fuel Debris Properties (Development of Technology for Estimating Damage Conditions of the Reactor Pressure Vessel)).



## 1. Grasping the condition of the sampled area

Blue: Additional analysis

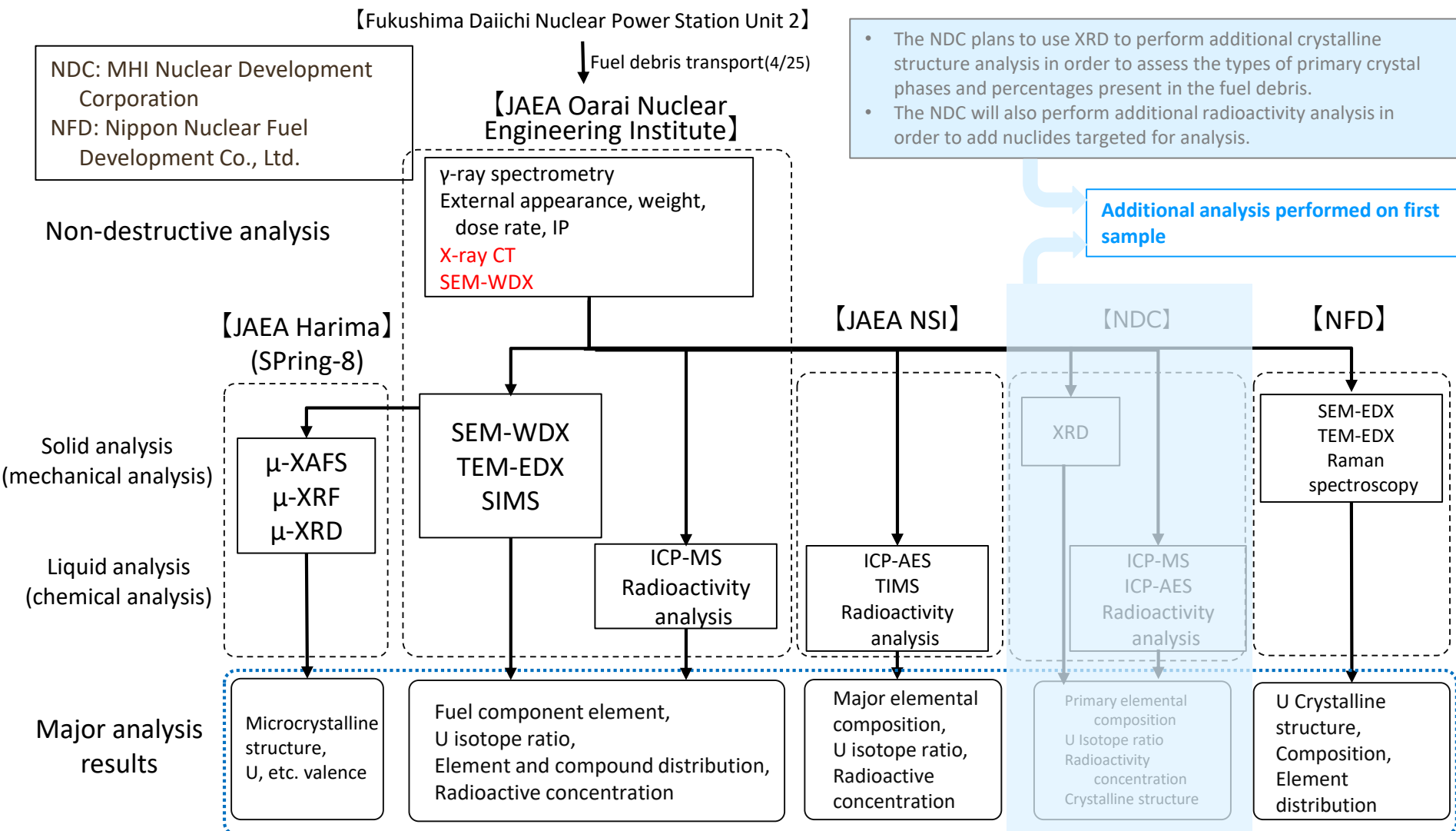
Refer the list of abbreviations at the end of the document for abbreviations of analysis methods

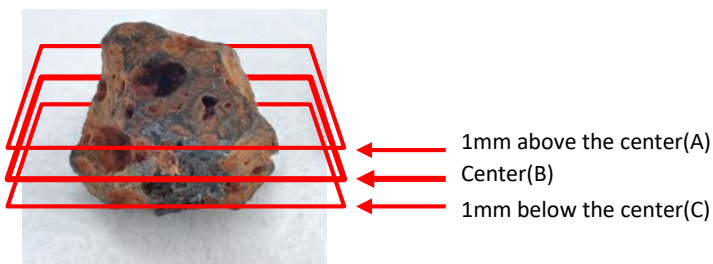
Analysis items	Analysis methods	Evaluation details	Examples of major applications for decommissioning
Basic information • External appearance, weight • Dose rate • Density distribution	• Exterior, weight, dose rate measurement • Imaging plate (IP) • X-ray CT	Organization of basic information	Basic information to review retrieval (existence and mount of pores, etc.)
Element content (elemental composition)	• ICP-MS, ICP-AES	Content of fuel components Origin of major components	Basic information to review safety measures at retrieval, such as criticality evaluation, and storage methods
Isotope ratio	• TIMS • SIMS	U isotope ratio	
Element and compound distribution	• SEM-EDX, SEM-WDX • TEM-EDX • XRD	Evaluation of distribution of elements and compounds (including pores)	Basic information to review retrieval methods and tools (estimation of hardness, toughness, etc.)
Radioactive concentration	• γ-ray spectrometry • α-ray spectrometry • β-ray spectrometry • Liquid scintillation counter, etc.	Accompanied condition of U with focal nuclides Amount of radioactivity of nuclides targeted for analysis	Information to review technology development for non-destructive measurement at fuel debris retrieval Information needed to deliberate treatment/disposal

## 2. Estimation of formation process of fuel debris

Analysis items	Analysis methods	Evaluation details	Examples of major applications for decommissioning
Crystal structure and composition of phases including U	• SEM-EDX, SEM-WDX • TEM-EDX • Raman spectroscopy • XRD • μ-XAFS                      • μ-XRF • μ-XRD	Estimation of temperature and atmosphere when U particles, etc. are formed Oxidation state of U, etc.	Precise estimated drawing of the condition inside the core to review retrieval methods and internal investigation

- Now that non-destructive analyses using x-rays, CT and SEM-WDX have been completed, those results will be reported.
- After reviewing batching and the amounts required for analysis, the second sample will be analyzed by four agencies.
- The NDC will use the first sample to conduct additional analysis.
- Analysis and result compilation is expected to take approximately one year at this point time.

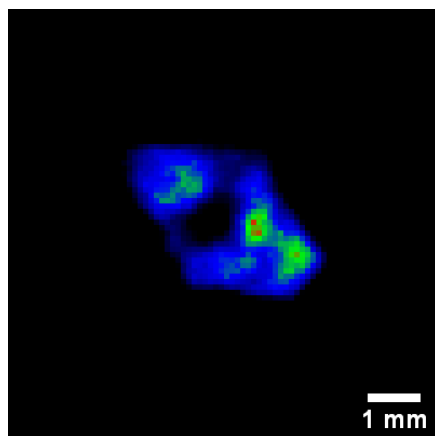




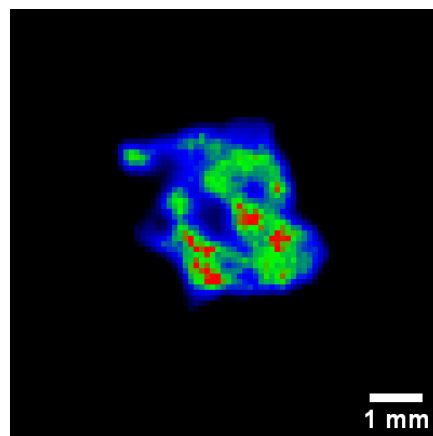
External appearance of fuel debris sample  
(X-ray CT image position)

## 【 Measurement method 】

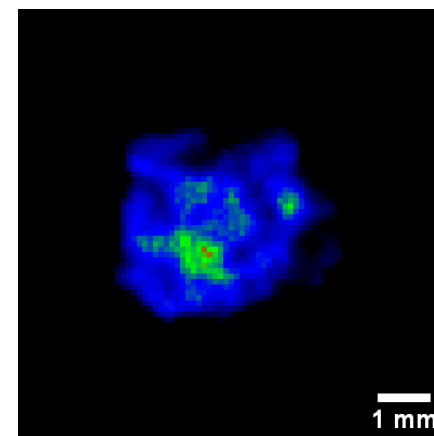
- Approx. 20 images are obtained by performing imaging at 0.2mm pitch vertically with the sample stored in a polypropylene container.



1mm above the center (A)

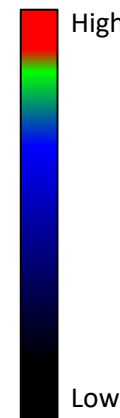


Center (B)



1mm below the center(C)

CT value



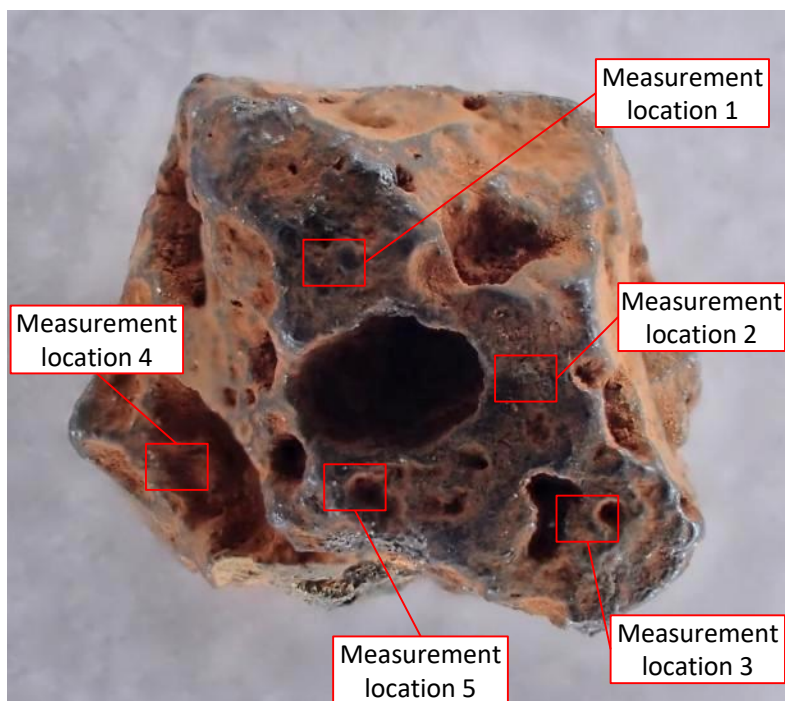
※CT (Computed Tomography) image is created from the X-ray transmission photography data

X-ray CT image of the fuel debris sample

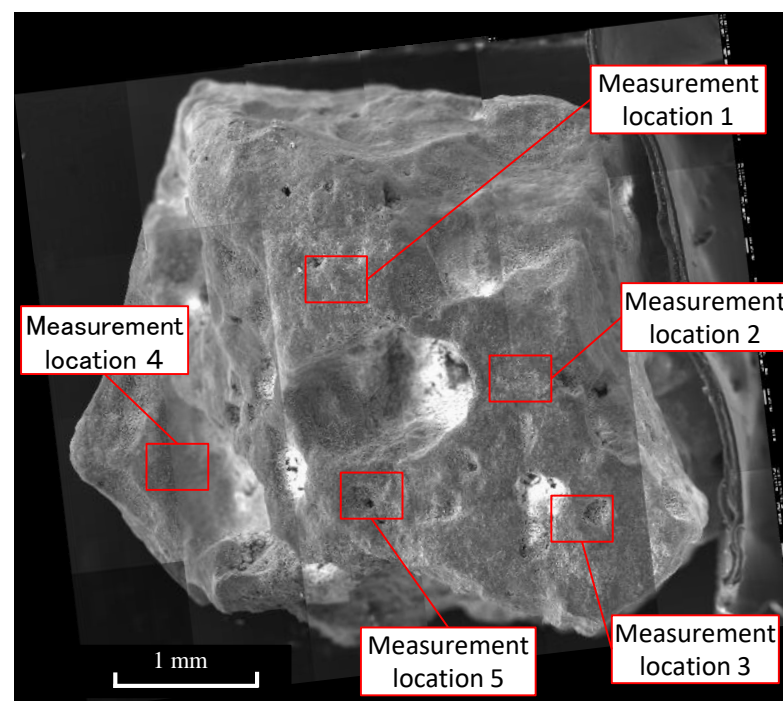
- Color-coded by CT value (correlation with density value) to grasp high-density areas and low-density areas.
- As with the first sample, low CT value regions (black: low-density) assumed to be pores are widely distributed throughout.
- Volume calculated from x-ray CT scans was approximately  $0.03\text{cm}^3$  \*. \*Under detailed review

- In order to review the policy for detailed analysis of the sample, element distribution of the sample surface was determined with SEM-WDX area analysis.
  - 5 measurement locations were selected away from each other, in order to obtain extensive information of the sample surface (refer to measurement locations 1~5 in the photos below).
  - Six elements were targeted for measurement during elemental mapping (U, Fe, Ni, Cr, Zr, O) at all measurement locations. Furthermore, point analysis spectrums were obtained from the center of each measurement location (each measurement field) and if any elements other than the aforementioned were detected, they were to be subjected to elemental mapping (However, no elements other than the aforementioned six elements were observed in this sample)

Photo of external appearance



SEM Observation results



Observations were taken after depositing beryllium vapor on the surface of the sample for conductive staining

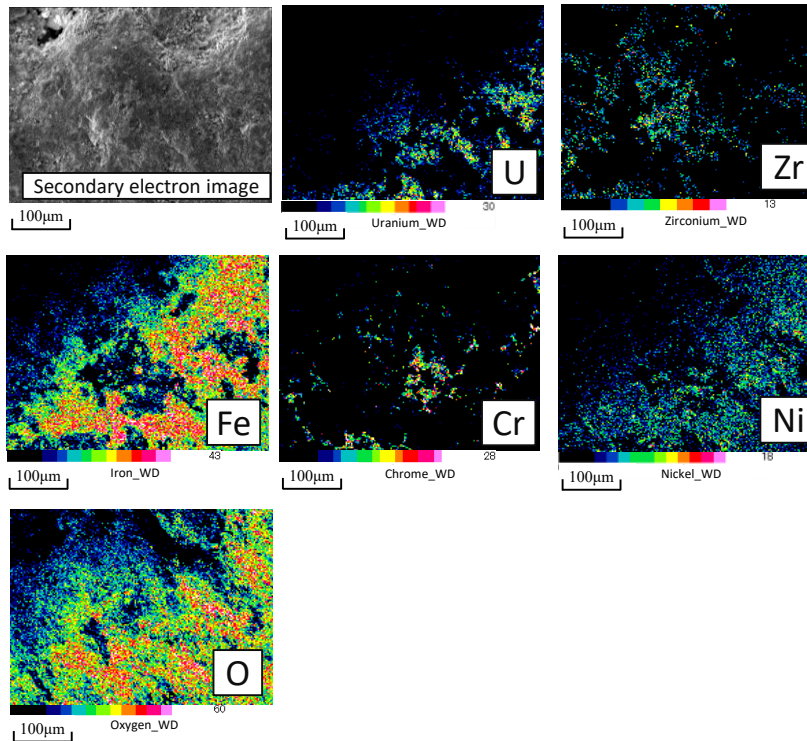
Measurement locations of SEM-WDX area analysis of the fuel debris sample surface



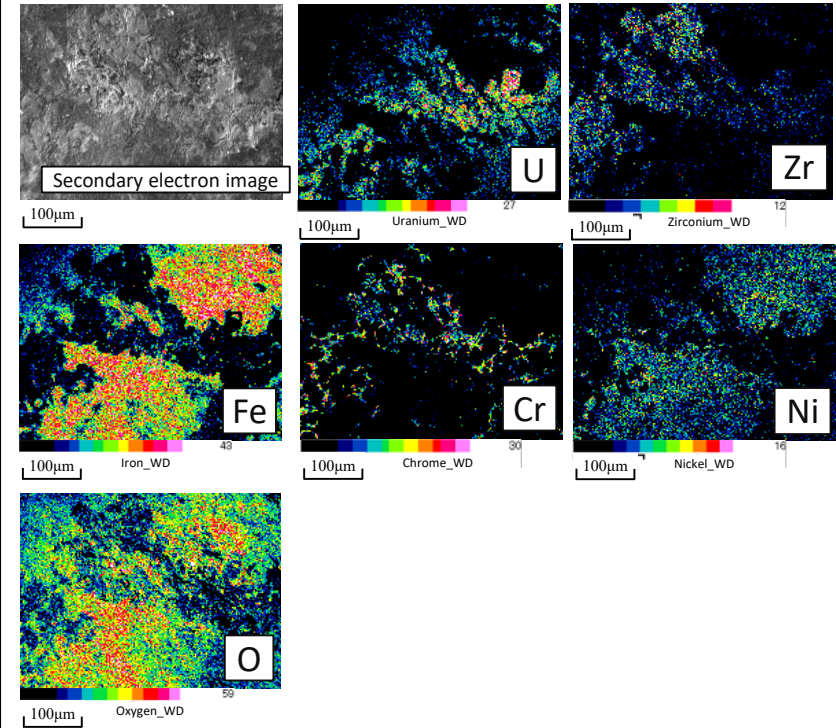
## WDX area analysis result

Note) The colors toward the right side of the legend indicate more amount of the element. Content cannot be compared among elements.

### Measurement location 1

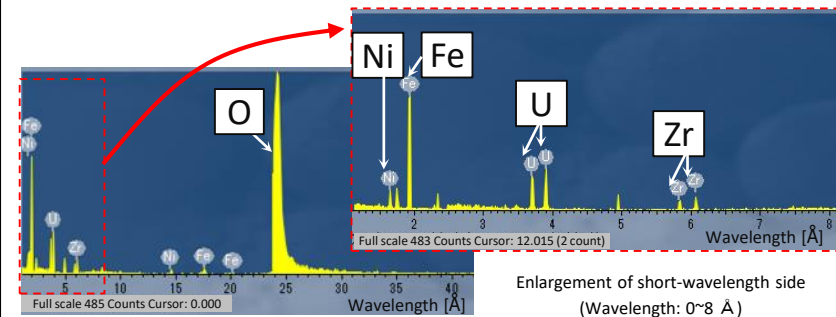
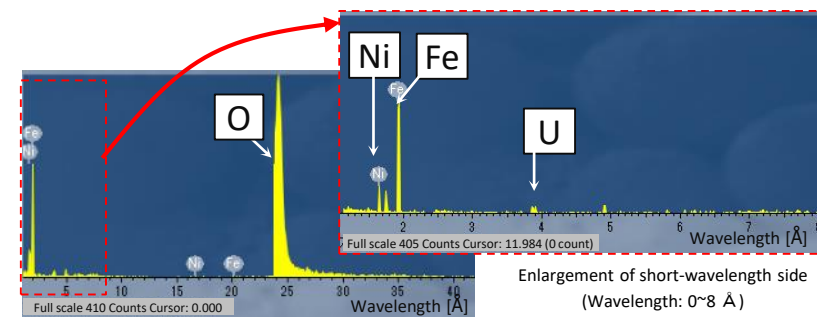


### Measurement location 2



## Reference) WDX point analysis spectrum

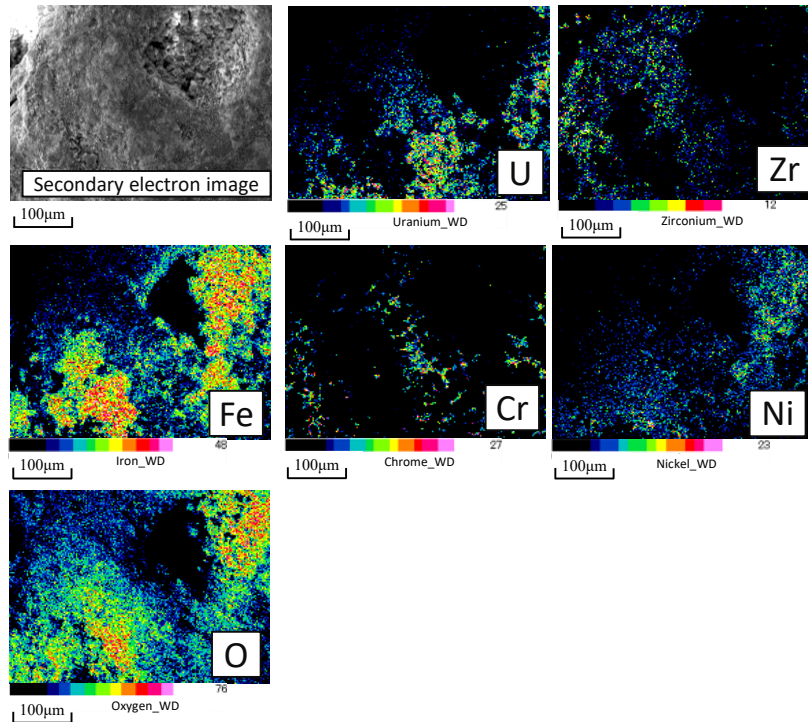
Note) Center of the field of view of area analysis measurement is measured



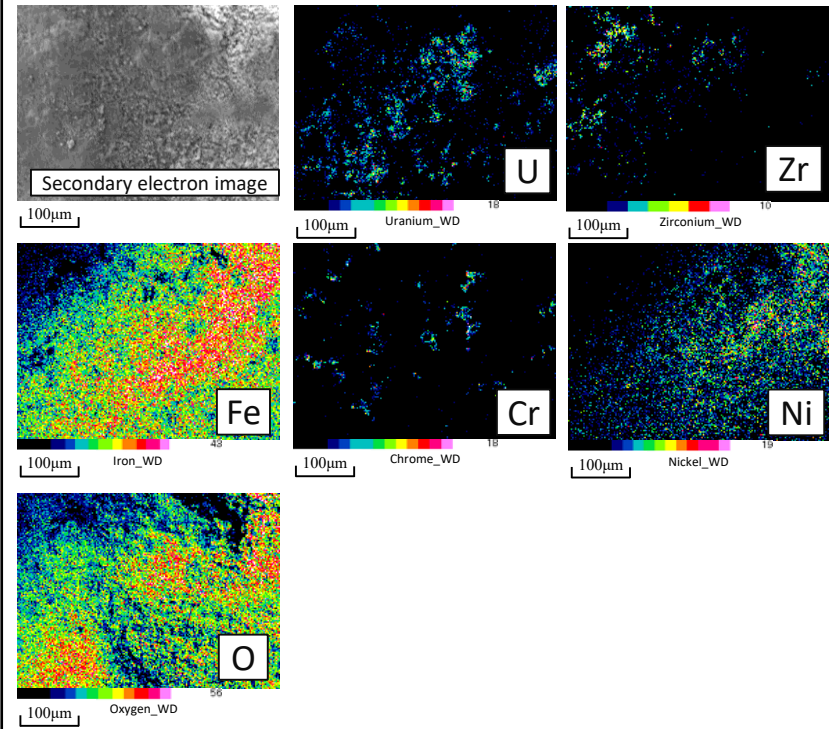
## WDX area analysis result

Note) The colors toward the right side of the legend indicate more amount of the element. Content cannot be compared among elements.

### Measurement location 3

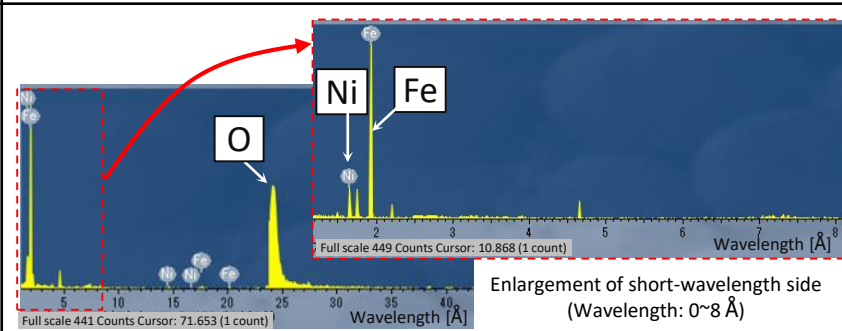
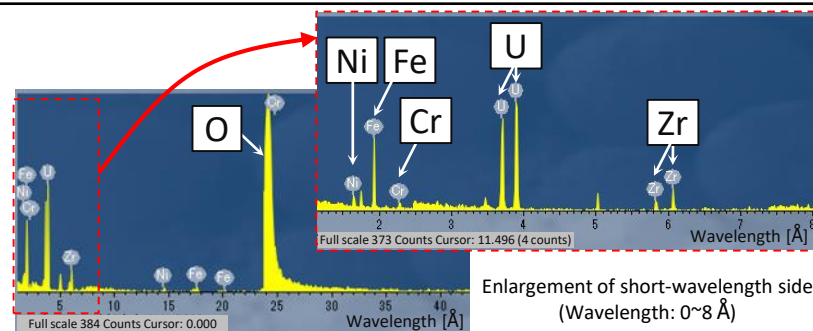


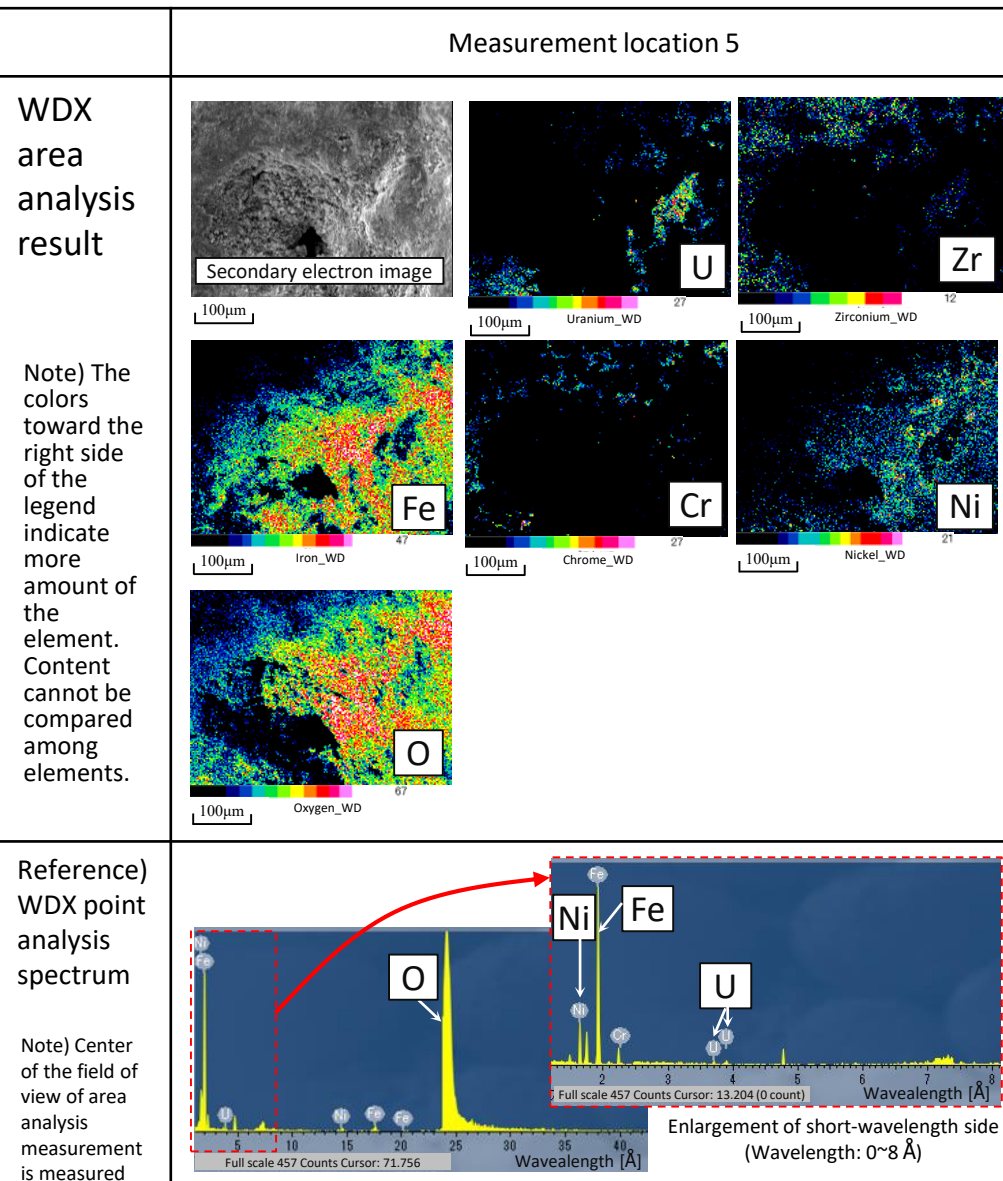
### Measurement location 4



## Reference) WDX point analysis spectrum

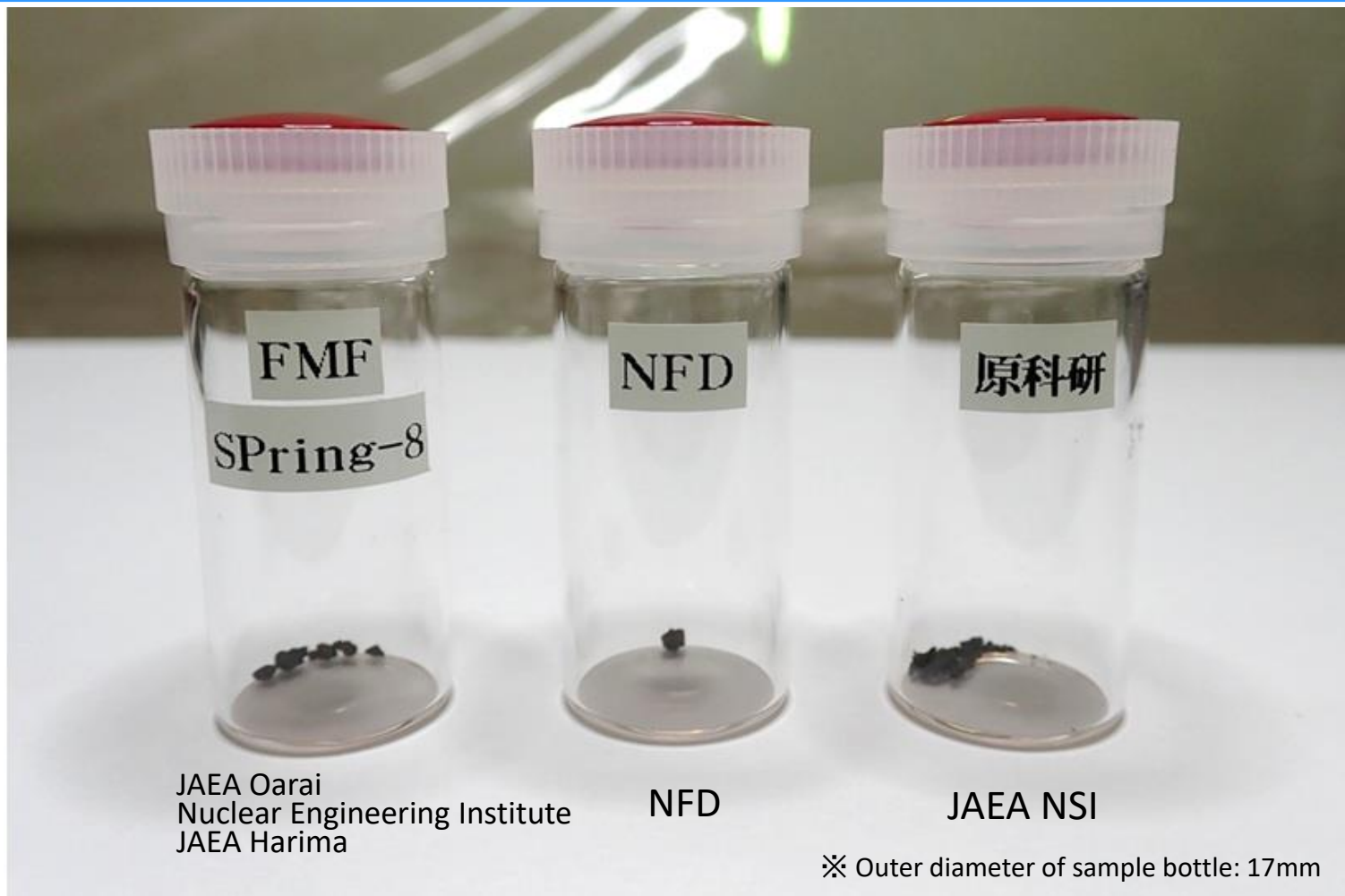
Note) Center of the field of view of area analysis measurement is measured





- As with the first sample, U is widely distributed over the surface
  - U, Zr, Fe, Cr, Ni and O (6 elements) were detected in all fields
  - Si, Ca and Al, etc., which were detected in the first sample along with the aforementioned six elements, were not detected this time.
- ⇒ It is assumed that the primary six elements originate from fuel elements (U), cladding tube/CB elements (Zr) and other structural components (Fe, Cr, Ni).





- Samples could be batched using the method employed for the first sample (crushing and pulverization with a stainless steel rod (approximately 250g)) and the fragments were sorted by hand.
- Samples were split amongst the four agencies in accordance with sample size and the amounts required for analyses.

## 【 Summary 】

### ○ X-ray CT results

- As with the first sample, shape and CT values overall were heterogeneous and there is a wide distribution of pores.

### ○ SEM-WDX results

- U is widely distributed on the sample surface as with the first sample.
- Differing from the first sample, only U, Zr, Fe, Cr, Ni and O (6 elements) were detected in all fields and Si, Ca and Al, etc., were not detected.
- The major 6 elements are estimated to be derived from the fuel elements (U), cladding/CB elements (Zr), and other structural material elements (Fe, Cr, Ni).

### ○ Fractionation results

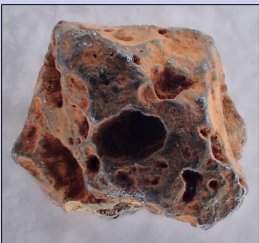

- The sample could be crushed and fractionated by hand employing the same method as used for the first sample. Samples were sent to JAEA NSI, the NFD and JAEA Harima (Spring-8), for detailed analysis (solid and liquid analysis), which will also be performed at the JAEA Oarai Nuclear Engineering Institute.

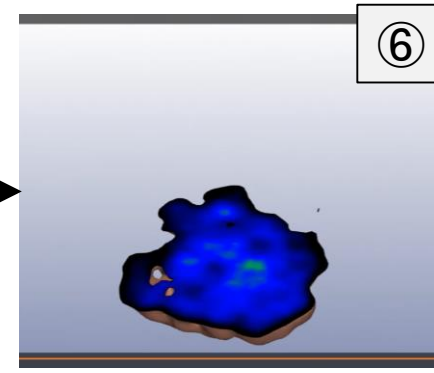
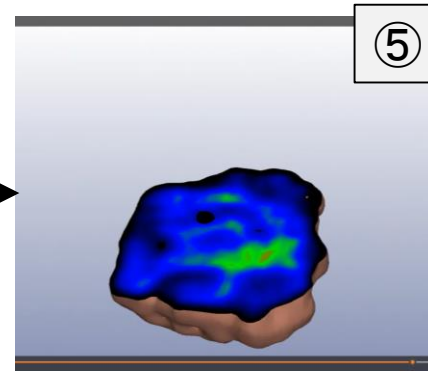
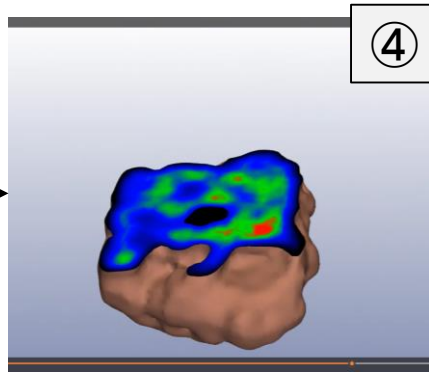
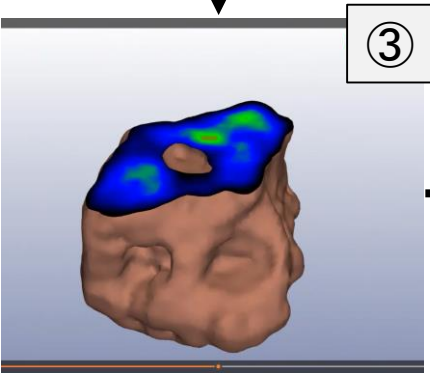
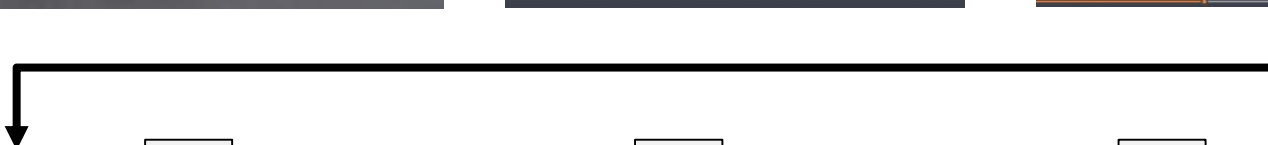
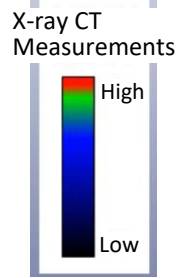
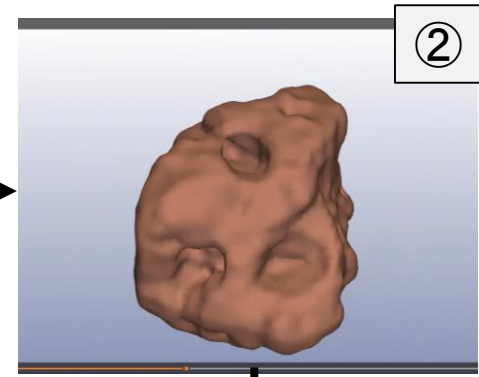
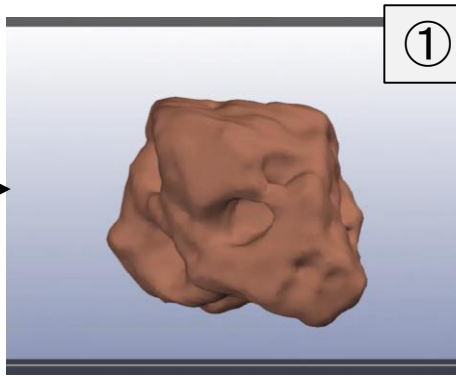
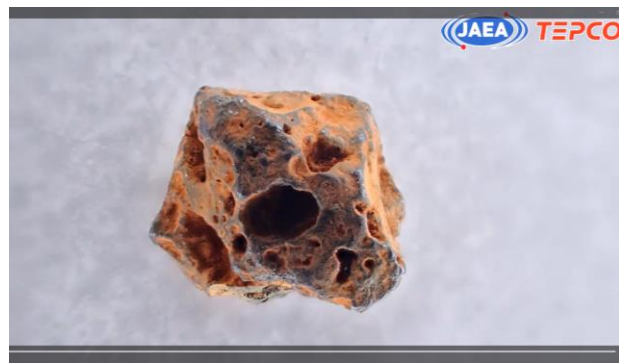
The above are the results of analysis of a few points on the sample surface, and evaluation will need to be based on the results of further detailed analysis. However, compared to the first analysis, elements such as Si, Ca, and Al derived from the thermal insulation layer and seawater were not detected, which suggests that the material inside the pressure vessel may have contributed relatively significantly to the generation process.

## 【 Future plans 】

- At this point time we expect detailed analysis (solid and liquid analysis) to take approximately one year after which the results will be compiled.



	2 <sup>nd</sup> sample	[Reference] 1 <sup>st</sup> sample	Notes
Outer appearance	 <p>Chestnut-brown overall with black regions and holes on the surface.</p>	 <p>Reddish brown overall with glossy black regions on the surface</p>	The difference in color can be attributed to iron oxidation. A detailed analysis will be performed to confirm.
Mass	0.187g (Total mass of the sample)	0.693g ✖	—
Size	Approx. 5mm×Approx. 4mm	Approx. 9mm×Approx. 7mm	—
Dose rate	Approx. 0.3mSv/h (γ-rays)	Approx. 8mSv/h (γ-rays)	—
γ spectrum	<sup>241</sup> Am, <sup>154</sup> , <sup>155</sup> Eu, <sup>125</sup> Sb, <sup>137</sup> Cs, <sup>60</sup> Co detected	<sup>241</sup> Am, <sup>154</sup> , <sup>155</sup> Eu, <sup>125</sup> Sb, <sup>137</sup> Cs, <sup>60</sup> Co detected	Same results
X-ray CT	CT value is heterogeneous Distribution of low-density regions assumed to be pores Volume: Approx. 0.03cm <sup>3</sup> ✖	CT value is heterogeneous Distribution of low-density regions assumed to be pores Volume: Approx. 0.1cm <sup>3</sup>	Same results ✖Under detailed review
Surface observation SEM-WDX	U, Zr, Fe, Cr, Ni, O detected Si, Ca, Al, etc. were not detected	Si, Ca, Al, etc. detected in addition to U, Zr, Fe, Cr, Ni, O	2 <sup>nd</sup> sample was more homogenous
Fractionation stutas	Crushed by hand with a stainless steel rod	Crushed by hand with a stainless steel rod	—
Detailed analysis	To be implemented by four agencies going forward	Underway at five agencies	—



- 3D animation was created based on X-ray CT images (20 images in total).
- Distribution of X-ray CT values of the interior can be observed in cross section.
- A version with a photo of the exterior of the sample pasted to the surface of the fuel debris will be created and posted on JAEA Fukushima Research and Engineering Institute Website.

【URL】 <http://fukushima.jaea.go.jp/debris>

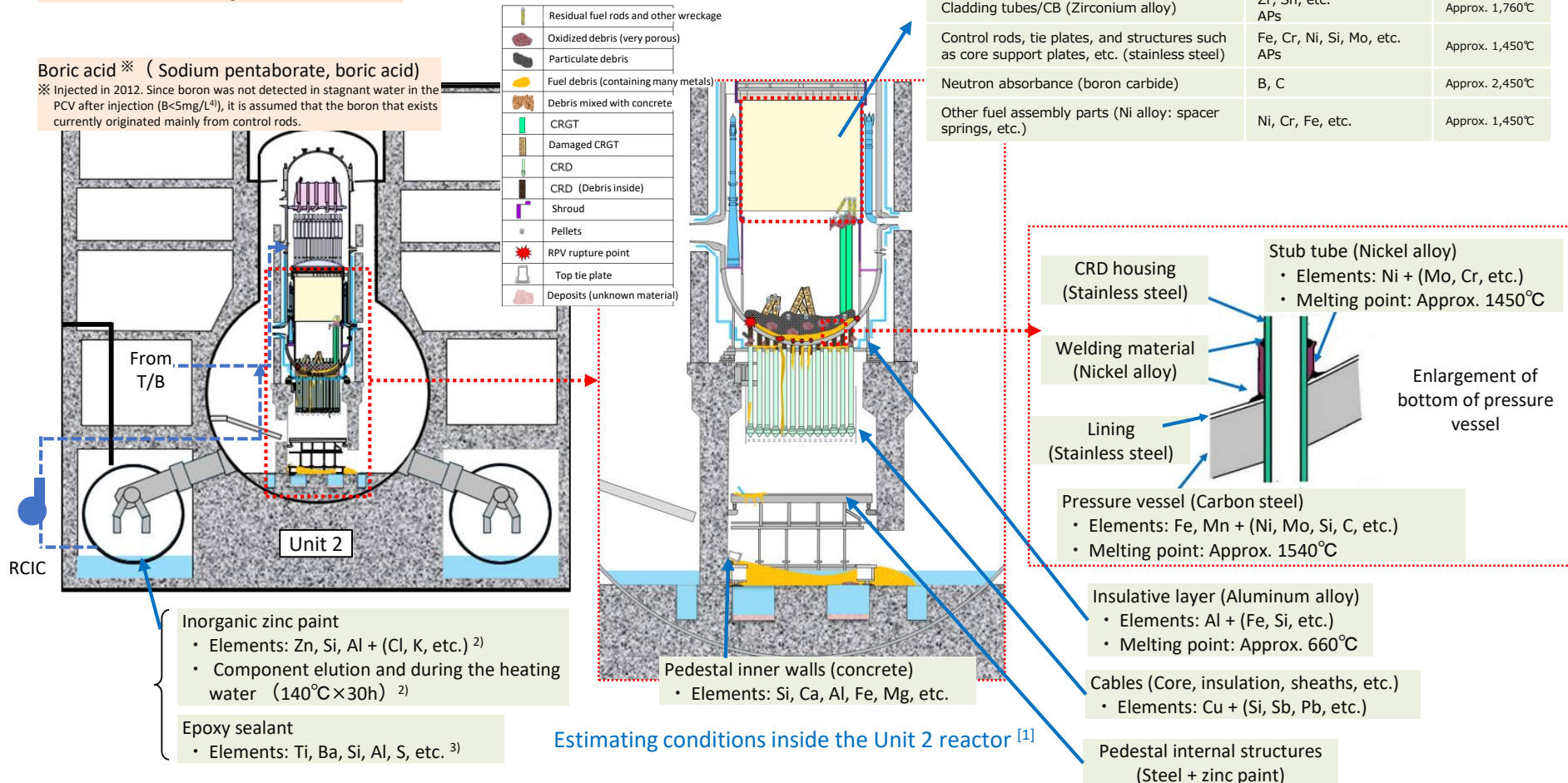
- Materials that might have been caught up in the formation process of the fuel debris sample due to the reaction to high temperatures during the accident and when the fuel debris migrated were identified and are useful for hypothesizing the fuel debris formation process.

## Seawater

- Elements: Cl, Na, Mg, S, K, Ca, etc. <sup>1)</sup>

## Boric acid ※ (Sodium pentaborate, boric acid)

※ Injected in 2012. Since boron was not detected in stagnant water in the PCV after injection ( $B < 5 \text{ mg/L}^4$ ), it is assumed that the boron that exists currently originated mainly from control rods.



[1] Kirishima et al., J. Nucl. Sci. Technol. 52, (2015), 1240. 2) Nakamori, et al., Atomic Energy Society of Japan 2018 spring conference, 2M17.

3) TEPCO HD Fukushima Daiichi Nuclear Power Station Accident Analysis Review Meeting (28th) document 4-1. February 28, 2022 (SEM-EDX results)

4) IRID, JAEA, Decommissioning/contaminated water countermeasures team meeting/secretariat meeting (39th) document 3-4-4. February 23, 2017. (Analysis results of stagnant water in the PCV)

[1] JAEA, Decommissioning/contaminated water/treated water countermeasure project commenced in FY2023 (development of analysis/estimation technology for ascertaining the nature of fuel debris) 3. Development of technology for estimating RPV damage and the migratory behavior of fuel debris inside the PCV-Final report-

The following 3 types of analysis are used to analyze the fuel debris sample and identify its characteristics and how it was formed.

## ● Non-destructive analysis

[Overview] Roughly grasp information, such as distribution of pores and high-density materials and contained components, without changing the state of the received sample as much as possible.

[Purpose] Obtain basic information of the sample, and confirm the presence or absence of components derived from nuclear fuel (uranium, radioactive nuclides, etc.) early on. Additionally, review how to specifically proceed with the analysis, such as which area to focus on in the solid analysis and liquid analysis to be conducted later on and which data to be obtained in what precision.

[Analysis methods] External appearance, weight, dose rate, IP, X-ray CT,  $\gamma$ -ray spectrometry, SEM-WDX (surface)

## ● Solid analysis

[Overview] Confirm what kind of state uranium, zirconium and other components from the reactor are in (what the coexisting elements are, whether it retains its pre-accident state, whether it is oxidized, etc.), by fractionating parts of the sample and observing its cross section in detail.

[Purpose] Obtain information on “how the sample was formed”, such as which materials reacted under what temperature or atmosphere\* to form the sample.

\*The synchrotron analysis of SPring-8, which was newly added after the previous report, is considered to enable more accurate estimation of the temperature and atmosphere at the time of the accident, since more detailed data than the conventional observation method based on electron microscopes can be obtained such as three-dimensional distribution of elements in the sample and valence of uranium.

[Analysis methods] SEM-EDX, SEM-WDX, TEM-EDX, SIMS, Raman spectroscopy,  $\mu$ -XAFS,  $\mu$ -XRF,  $\mu$ -XRD

## ● Liquid analysis

[Overview] Fractionate part of the sample and dissolve it in acid to measure the elements and nuclide content in the resulting dissolving solution.

[Purpose] Obtain necessary information to review the process to safely retrieve and stably store fuel debris, such as uranium isotope ratio and radioactive nuclide concentration.

[Analysis methods] ICP-MS, ICP-AES, TIMS,  $\gamma$ -ray spectrometry,  $\alpha$ -ray spectrometry

Continuing the series of analyses will gradually identify the characteristics of fuel debris deposited in the core and contribute to safety evaluation and rationalization for fuel debris retrieval and storage.

[1] JAEA, Fuel debris sample non-destructive analysis results, Decommissioning, Contaminated Water, Treated Water Countermeasures Team Secretariat Meeting (138<sup>th</sup> meeting), May 29, 2025

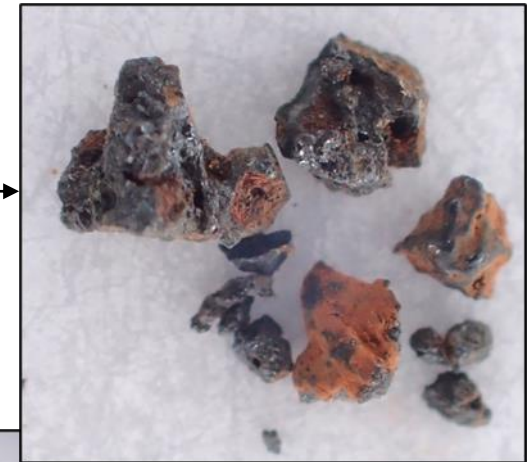
Analysis method abbreviation	Analysis method name	Analysis method overview
ICP-AES	Inductively coupled plasma atomic emission spectroscopy	Qualitative and quantitative analysis method of elements by introducing atomized samples into high-temperature plasma and obtaining element-specific spectra by spectroscopy of the issued light.
ICP-MS	Inductively coupled plasma mass spectrometry	Method of measuring the concentration of elements and its isotopes by introducing atomized samples into high-temperature plasma, ionizing elements in the sample and measuring the number of ions in ion mass-to-charge ratio ( $m/z$ ) by mass spectrometry.
TIMS	Thermal ionization mass spectrometry	Method of measuring the concentration of elements and its isotopes by applying samples onto metal filament, ionizing the atoms by heating under vacuum and measuring the number of ions in ion mass-to-charge ratio ( $m/z$ ) by mass spectrometry.
IDMS	Isotope dilution mass spectrometry	Method for measuring the elemental mass (concentration) of a specimen targeted for analysis (analyte) by adding a known amount of a rare isotope with a totally different isotopic composition and measuring changes to the isotopic composition mass of the analyte before and after the changes and the amount of standard sample added. Isotopic composition is measured through mass spectrometry.
SEM	Scanning electron microscope	Device that can observe the sample surface by irradiating the surface with electron beams, and can also analyze elements by attaching an X-ray analyzer.
EDX	Energy dispersive X-ray spectroscopy	Method of elemental analysis and compositional analysis by detecting characteristic X-rays generated by electron irradiation and categorizing them by the energy of characteristic X-rays.
WDX	Wavelength dispersive X-ray spectroscopy	Method of elemental analysis and compositional analysis by detecting characteristic X-rays generated by electron irradiation and performing spectroscopy at the wavelength of characteristic X-rays.
TEM	Transmission electron microscope	Method of imaging electrons transmitted through the sample and scattered electrons for observation under high magnification by irradiating thinned samples with electron beams, and also conducting elemental analysis by attaching an X-ray analyzer. Crystal structure can also be obtained from the diffraction image.
SIMS	Secondary ion mass spectrometry	Method of measuring the concentration of elements and its isotopes by measuring the secondary ions generated by irradiating the sample surface with a beam of ions with a mass spectrometer and measuring the number of ions in ion mass-to-charge ratio ( $m/z$ ) by mass spectrometry.
Raman spectroscopy	Micro raman spectroscopy	Method of obtaining properties such as molecular structure, temperature, stress, electrical properties, orientation and crystallinity by irradiating the sample surface with light and dispersing Raman scattering light. Information on chemical form of micro-regions on $\mu\text{m}$ order can be obtained by combining Raman spectroscopy with conventional optical microscopes.



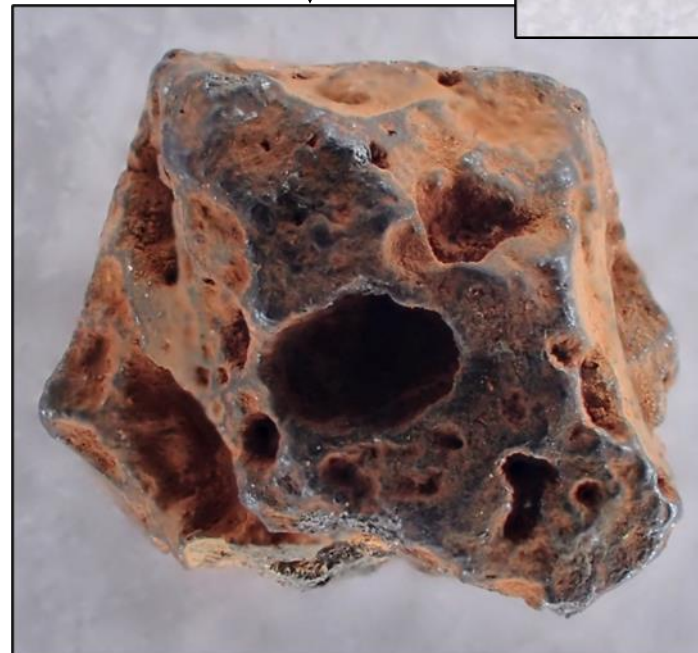
Analysis method abbreviation	Analysis method name	Analysis method overview
X-ray CT	X-ray computed tomography	Method of obtaining density distribution of the sample interior by irradiating the sample with X-rays, capturing the transmitted X-ray intensity by a computer and scanning it three-dimensionally. Distribution of phases of different density can be obtained.
XAFS	X-ray absorption fine structure spectroscopy	Method of analyzing the internal structure of materials at the molecular and atomic level by irradiating the sample with X-rays and precisely observing the absorbed X-ray energy.
XRF	X-ray fluorescence spectroscopy	Method of qualitative analysis of content of constituent elements by measuring the wavelength and energy of X-rays (X-ray fluorescence) generated according to the substance by irradiating the sample with X-rays
XRD	X-ray diffraction analysis	Method of analyzing the crystal structure, crystal orientation, crystal lattice size, etc. of the object by irradiating the sample with X-rays and measuring the resulting X-rays (diffracted X-ray)
IP	Imaging plate	Radiation image measuring instrument that detects radiation energy as stimuable luminescence. Dose distribution of the sample can be obtained.



(Small fragments)

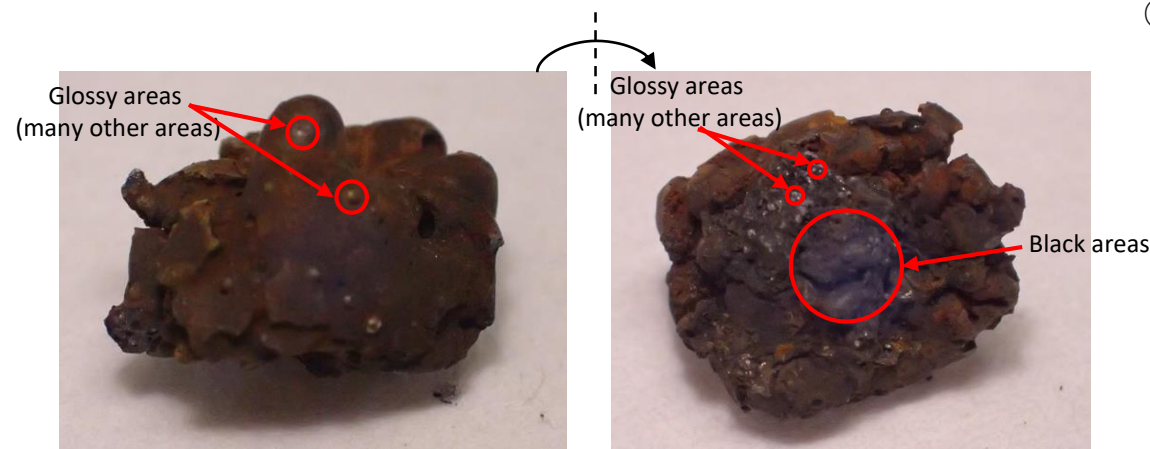


External appearance of fuel debris sample  
(color samples (color separation guide) with scale)



Enlarged photo of External appearance of fuel debris sample  
(photographed from approx. 45 degrees diagonally)





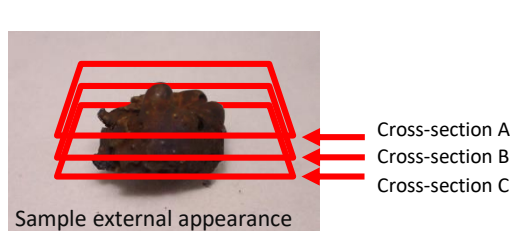
Size: Approx. 9mm×Approx. 7mm (compared with scale)

Figure 1 External appearance of fuel debris sample (after arrival at JAEA Oarai)

## ○ External appearance, mass, dose rate

- The received sample was reddish-brown overall with an indefinite shape. Black and glossy areas could be seen on the surface. (Figure 1)
- Mass: 0.693g
- Dose rate: Approx. 8mSv/h (γ-rays) ※1

※1 An ionization chamber was used to measure the sample while it was still inside a polypropylene container (at a distance of 1~2 cm from the sample))  
IP imagery (dose distribution) could not show an accurate distribution due to the high dose rates and small size of the sample.



### 【Measurement method】

- Images taken vertically at 0.2 mm intervals after putting the sample into a polypropylene canister. A total of 38 images were obtained.
- CT values (showing the correlation to density) were color-coded in order to ascertain areas of high density and low density.

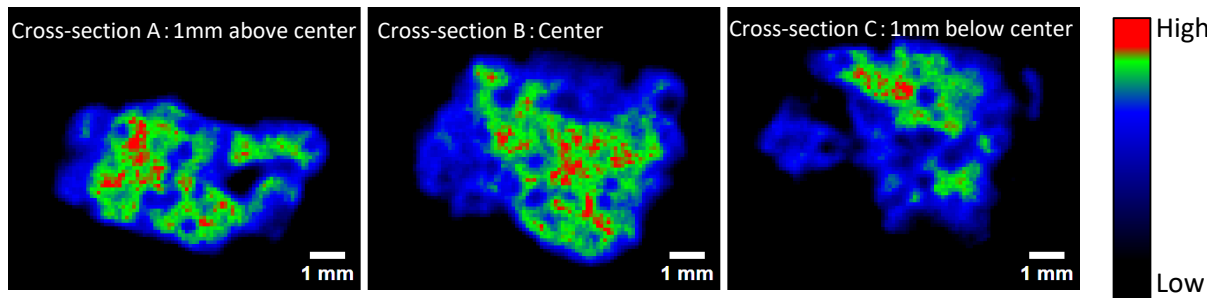


Figure 2 Fuel debris sample x-ray CT images

## ○ X-ray CT

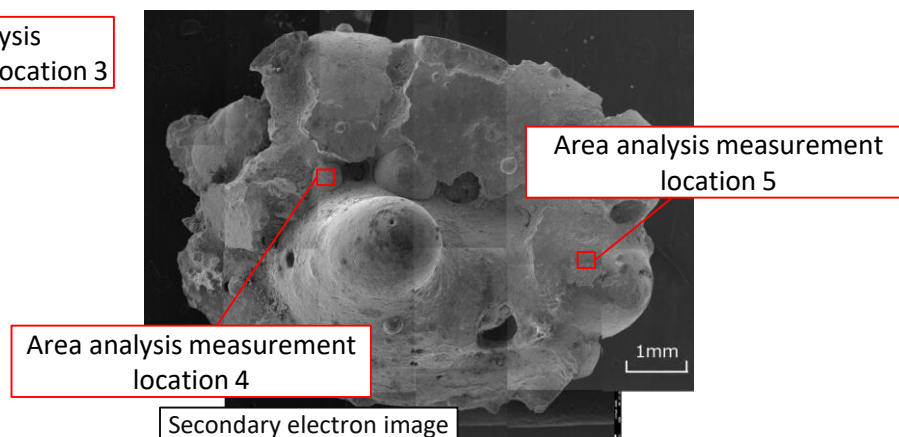
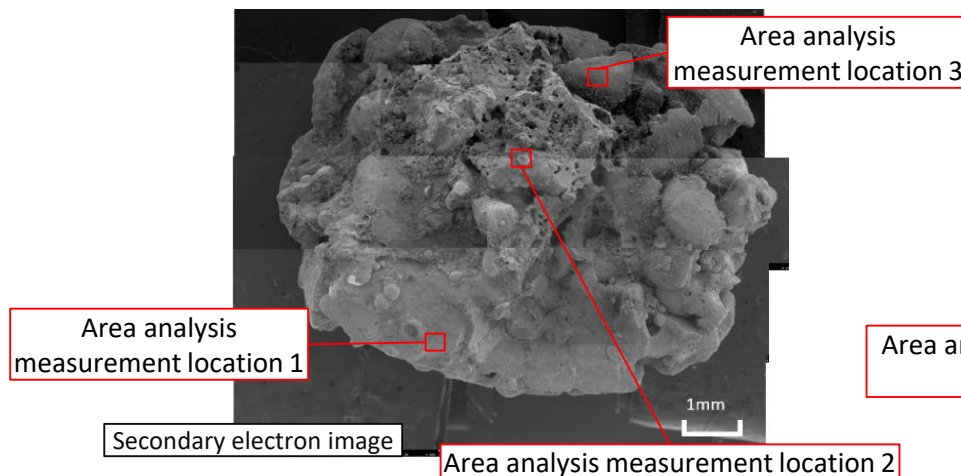
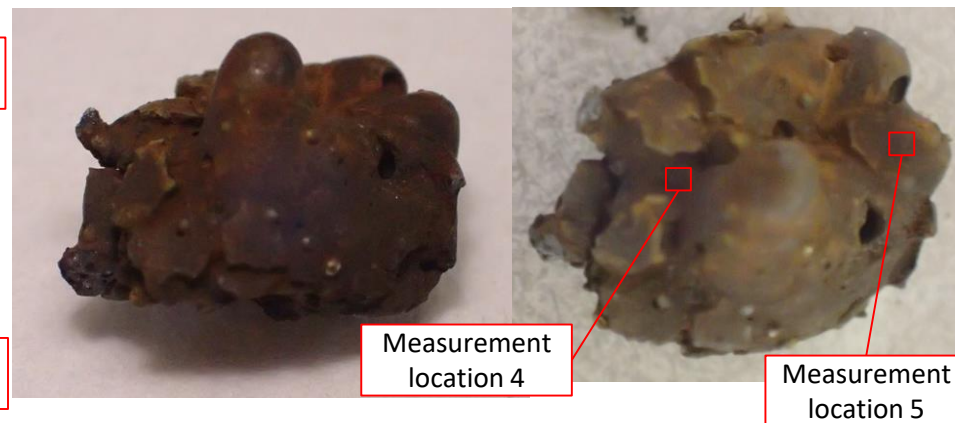
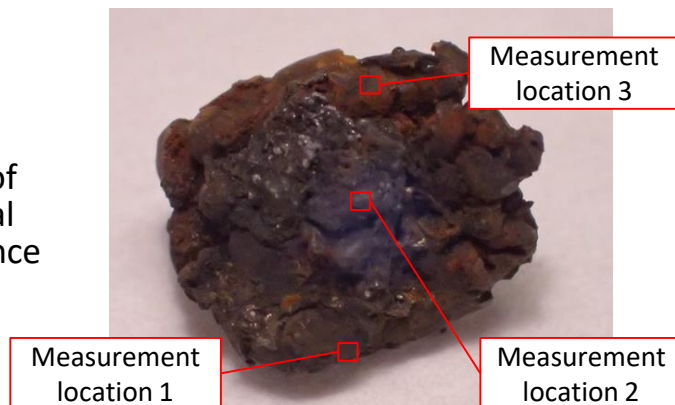
- Relatively high-density areas (red) and pores (black) profound inside the sample. (Figure 2)
- Results calculated from the x-ray CT image found the volume ※2 to be approx. 0.1cm<sup>3</sup>.  
→ Density estimated to be approx. 7g/cm<sup>3</sup> from the aforementioned mass and volume.

※2 Includes internal pores but excludes surface pores



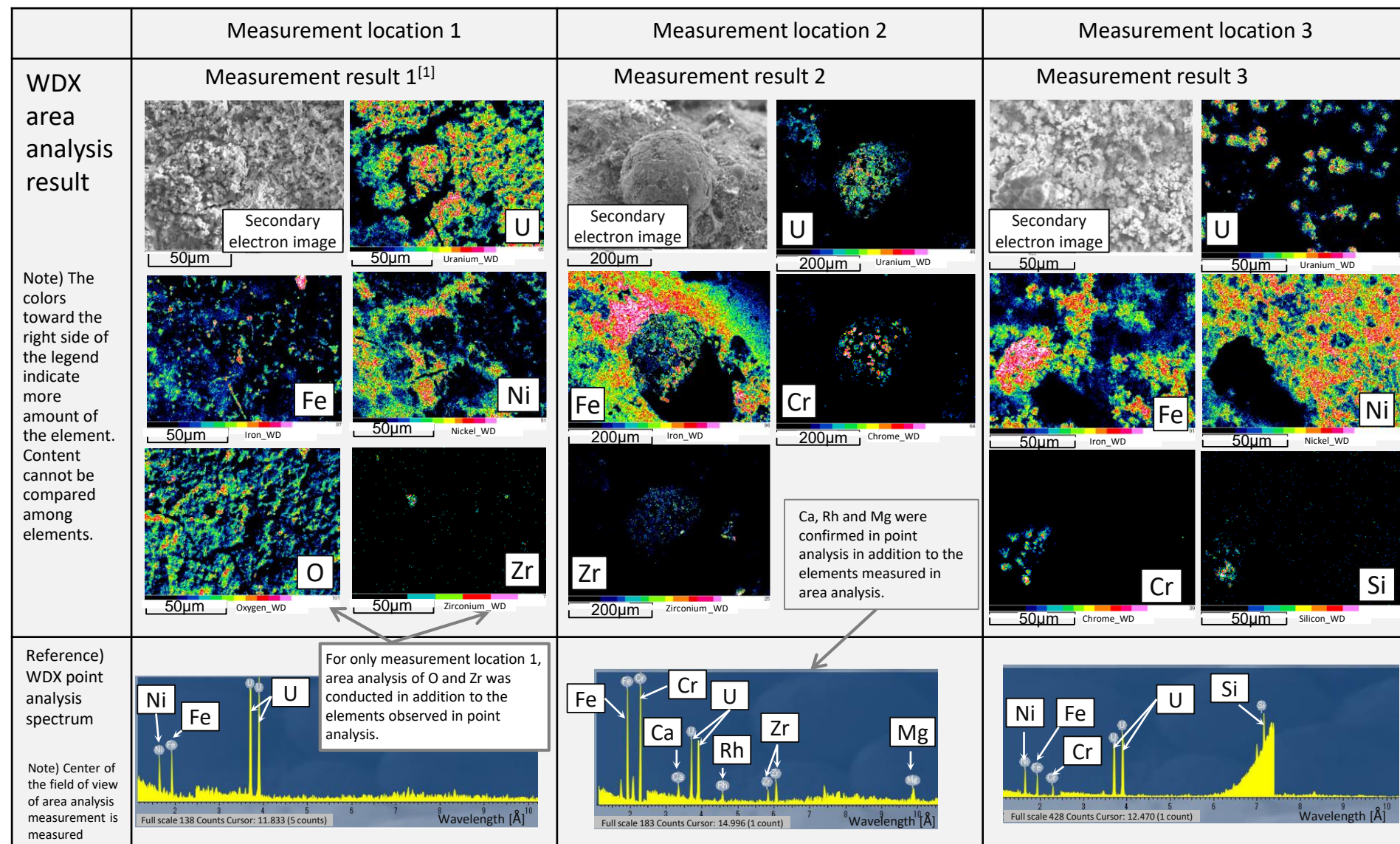
- In order to review the policy for detailed analysis of the sample, element distribution of the sample surface was determined with SEM-WDX area analysis.
  - 5 measurement locations were selected away from each other on the front and back sides of the sample, in order to obtain extensive information of the sample surface (see measurement locations 1-5 below; measurement location 1 is the same as the previous report).
  - Area analysis was conducted after point analysis.
  - In addition to U, Fe (common to all measurement locations), major elements that were identified with the point analysis spectrum were added as elements to be measured in area analysis (the number of elements to be measured per field of view is limited to 4-5 elements in order to secure the analysis period).

Photo of external appearance

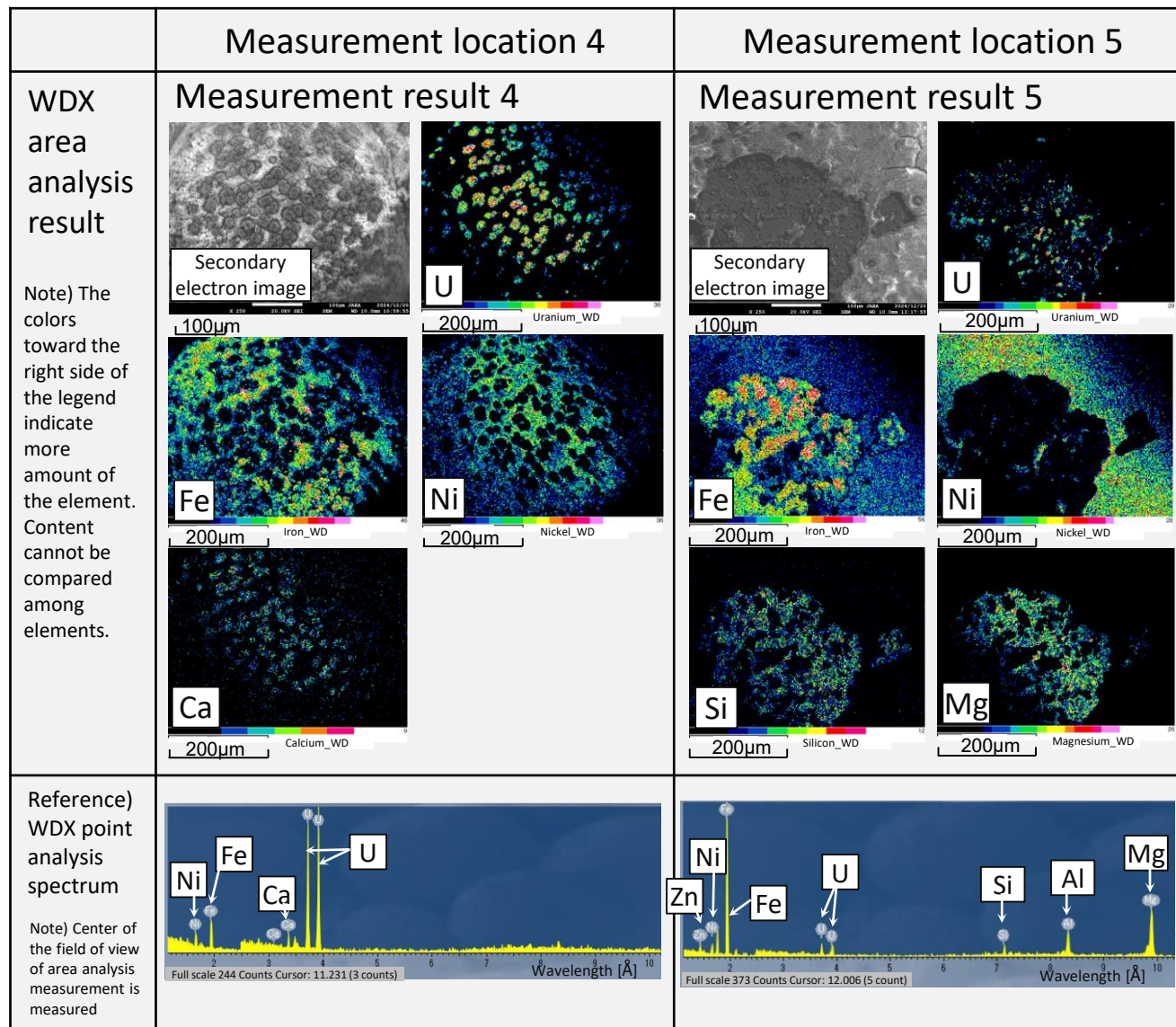


Measurement locations of SEM-WDX area analysis of the fuel debris sample surface





SEM-WDX measurement results of fuel debris samples (Measurement location 1-3)



- U and Fe were observed on all fields of view. However, the location of U does not match with the location of Fe. Some fields of view also suggested less U and more Fe (Measurement location 5).

⇒ The fuel debris sample is heterogeneous, but U is considered to be widely distributed at least on the sample surface.

Zn and Al were confirmed in point analysis in addition to the elements measured in area analysis.

## SEM-WDX measurement results of fuel debris samples (Measurement location 4-5)